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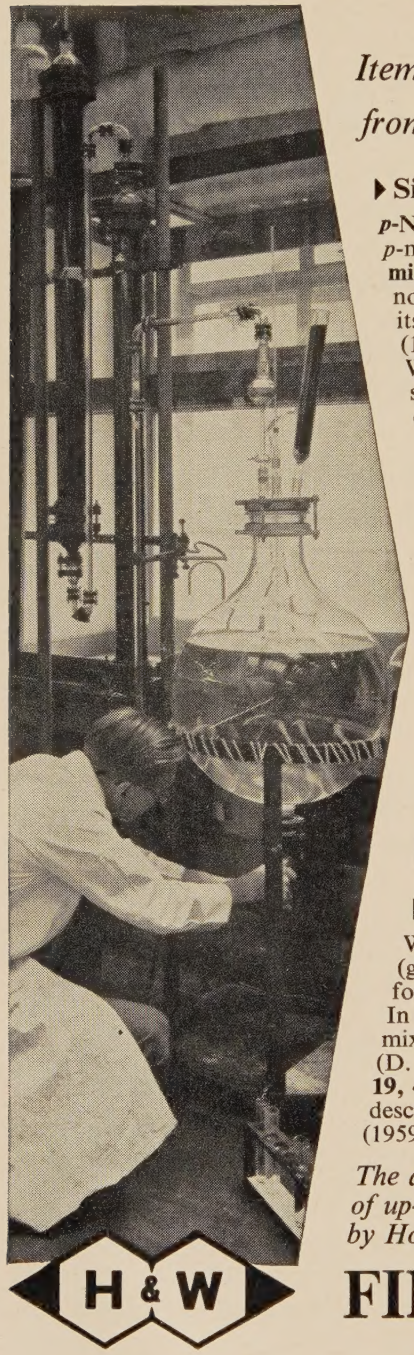

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from our laboratory notebooks

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In the solid state calcium oxide can be detected in mixtures of magnesium, strontium and barium oxides. (D. Goldstein and C. Stark-Meyers, *Anal. Chim. Acta*, **19,** 437 (1958)). Its use as a complexometric indicator is described by Goldstein in *Anal. Chim. Acta*, **21,** 339 (1959).

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

General reviews of progress, reagents and methods of general application.

1338. **The dawn of micro-analysis.** C. Duval Micro-anal. Res. Lab., 11 rue Pierre Curie, Paris, France). *Mikrochim. Acta*, 1960, (5–6), 630–635 in French).—A historical review of 18th and 19th entury micro-analytical researches, with 36 refer-nces. J. P. STERN

1339. **The origins of colorimetric analysis.** F. zabadváry (Inst. Gen. Chem., Tech. Univ., Budapest, Hungary). *Talanta*, 1960, **5** (2), 108–11 (in German).—The development of colorimetric nalysis during the period 1845 to 1880 is outlined, nd a list of authors of the period and 19 references re given. W. T. CARTER

1340. **Progress in microchemistry.** *Microchem.* ., 1960, **4** (3), 271–396.—The following review apers are included. **Progress in reduced-scale etermination of physical constants: 1959,** J. S. Viberley and H. W. Drake, 277–287 (57 ref.). **Progress in fractionation procedures. Differential migration methods: 1959,** A. G. Mistretta, 289–305 112 ref.). **Progress in quantitative inorganic nalysis: 1959,** R. Maurmeyer, 307–320 (58 ref.). **Progress in biochemical investigations. I. Nitrogen ompounds: 1959,** S. Natelson, 321–342 (202 ref.); **II. Determination of elements in biological systems: 959,** A. L. Levy, 343–352 (81 ref.). **Progress in lemental quantitative analysis: 1959,** A. Steyer-mark and B. E. McGee, 353–372 (165 ref.). **Pro-ress in organic functional group analysis: 1959,** T. S. Ma, 373–386 (57 ref.). **Progress in equipment nd tools: 1959,** H. J. Francis, jun., 387–396 104 ref.).

1341. **Determination of gases in metals.** Report of a Symposium organised by The Society for Analytical Chemistry, The Iron and Steel Institute and The Institute of Metals. *Iron and Steel Inst., Spec. Report* No. 68, 1960; 308 pp.—The following papers are included. **Sampling of liquid metals,** F. B. King, pp. 3–18. **The determination of oxygen in metals: a review,** W. T. Elwell, pp. 19–42. **The determination of gases in metals by vacuum fusion,** J. E. Still, pp. 43–63. **The determination of gases in metals by the semi-micro vacuum-fusion technique,** A. Parker, pp. 64–74. **An assessment of carrier-gas methods for the determination of gases in metals, with particular reference to steel,** E. A. Shanahan, pp. 75–92. **The determination of oxygen in beryllium by activation analysis,** R. F. Coleman, pp. 93–102. **Emission spectrometric determination of oxygen, nitrogen and hydrogen in metals,** V. A. Fassel, pp. 103–120. **The determination of oxygen and nitrogen in iron and steel by an isotope-dilution method,** M. L. Pearce and C. R. Masson, pp. 121–132. **The determination of nitrogen in metals: a review,** J. D. Hobson, pp. 151–182. **The determination of nitrides in metals,** H. F. Beeghly, pp. 183–191. **The determination of hydrogen in metals,** R. E. Eborall, pp. 192–218. **The determination of hydrogen in cast iron,** J. V. Dawson and L. W. L. Smith, pp. 219–228. **An improved carrier-gas technique for the determination of hydrogen in steel,** F. R. Coe and N. Jenkins, pp. 229–235. **X-ray emission analysis and the determination of gases in metals,** T. Mulvey, pp. 255–269. **Application of internal-friction measurements to the study of gases in metals,** G. M. Leak, pp. 270–295.

1342. **Use of nuclear magnetic resonance for analytical purposes.** N. M. Pomerantsev. *Zavod. Lab.*, 1960, **26** (8), 950–956.—A review, with 29 references, is presented. G. S. SMITH

1343. **Notes on the behaviour of diphenylcarbazone as an analytical reagent.** J. Minczewski and W. Żmijewska (Inst. Nucl. Res., Polish Acad. Sci., Warsaw). *Chem. Anal., Warsaw*, 1960, **5** (3), 429–433.—During the investigation of the Cr^{6+} - diphenylcarbazide (**I**) reaction it was found that diphenylcarbazone (**II**) in alcoholic soln. deteriorates on storage. The decomposition of 0·01M-**II** proceeded at a steady rate until about 75% of **II** was destroyed after 35 days, and after 3 months virtually no **II** remained in the soln. It was also confirmed that **II** in glacial acetic, formic and propionic acids produced nearly the same colour as that produced in the Cr^{6+} - **I** reaction, which would suggest that the colour is not due to a complex, but to a tautomeric form of **II**. P. BRYCH

1344. **The thermal properties of some amine tetraphenylboron salts. 8-Hydroxyquinoline and its derivatives.** W. W. Wendlandt, J. H. van Tassel and G. R. Horton (Dept. of Chem., Texas Technol. Coll., Lubbock, U.S.A.). *Anal. Chim. Acta*, 1960, **23** (4), 332–336 (in English).—Confirmation has been made of the existence of compounds having the composition A_2B, where B is the tetraphenylborate ion and A is 8-hydroxyquinoline, 8-hydroxy-2-methylquinoline, 5,7-dichloro-8-hydroxyquinoline, 5,7-dibromo-8-hydroxyquinoline or 8-hydroxy-5,7-di-iodoquinoline. The stability of the salts has been studied by thermogravimetric and by differential thermal analysis. The 5,7-dihalo-compounds show greatest stability. T. R. ANDREW

1345. **Complexans. IV. Synthesis and study of complexans of the triazine series.** R. P. Lastovskiĭ, I. D. Kolpakova and N. M. Dyatlova (All-Union Sci. Res. Inst. for Chem. Reagents, Moscow). *Zhur. Anal. Khim.*, 1960, **15** (4), 419–423.—4,6-Diamino-2-hydroxy-1,3,5-triazine-*NNN′N′*-tetra-acetic acid (**I**) and 2,4,6-triamino-1,3,5-triazine-*NNN′N′N″N″*-hexa-acetic acid (**II**) were synthesised. **I** forms compounds at pH 2·5 with Pb^{2+}, Cu^{2+}, Bi^{3+}, Cd^{2+}, Ni^{2+}, Mo^{6+} and Ti^{4+}, and also with

As^{3+} and Mn^{2+} at pH 4·4; at pH 9·35 it forms compounds with Pb^{2+}, Cu^{2+}, Cd^{2+}, As^{3+}, Co^{2+} and Mo^{6+}; and at pH 12 with Cu^{2+}, Cd^{2+}, Zn^{2+}, Ni^{2+} and Bi^{3+}; **II** forms compounds at pH 4·4 with Pb^{2+}, Cu^{2+}, Mn^{2+}, Mo^{6+} and Ti^{4+}, and also with La^{3+}, Tl^{+} and Zn^{2+} at pH 2·5; at pH 9·3 it forms compounds with Pb^{2+}, Cu^{2+}, As^{3+}, Mn^{2+}, Mo^{6+} and La^{3+}; and at pH 12 with Cu^{2+}, Cd^{2+}, Ni^{2+} and Mo^{6+}. For this study, differential polarography was used. The properties of **I** and **II** are compared with those of *m*-phenylenediaminetetra-acetic acid.

K. R. Cook

1346. **New mixed indicator for mercurimetry.** R. D. Glukhovskaya and N. A. Ugol'nikov. *Izv. Vyssh. Ucheb. Zavedenii, Khim. i Khim. Tekhnol.*, 1960, **3** (1), 49–51; *Ref. Zhur., Khim.*, 1960, (18), Abstr. No. 72,967.—The mixed indicator containing diphenylcarbazone (**I**) and 2-nitroso-1-naphthol (**II**) is used for the mercurimetric titration of halides. Both components are mercurimetric indicators, and also show oxidation - reduction properties (in alkaline soln. **I** and **II** are orange; in acid soln. **I** is colourless and **II** is yellow - green). The mixed indicator can thus be used to establish the required pH of the soln. at the beginning of the titration; if acid or neutral halide soln. are neutralised with alkali to an orange colour, and N-HNO_3 is added to a yellow - green colour, a suitable pH for the titration is obtained. At the end-point, the violet colour of the complex of Hg^{2+} with **I** and the orange colour of the complex of Hg^{2+} with **II** are superimposed, giving a cherry-red soln.; the colour change from yellow - green to cherry red is sharp and is easily seen, even with fairly dilute soln. (0·01N). The clearest colour change is obtained with 1 drop of 1% ethanolic **I** soln. and 1 drop of 3% ethanolic **II** soln. for each 5 or 6 ml of soln. A mixed indicator containing **I** and **II** in the same proportions gives similar results. The colour change is made sharper by titrating Cl^-, Br^- and I^- in an aq. ethanolic medium. C. D. Kopkin

1347. **Nitrophenylosazones and nitrophenylhydrazones as indicators for high pH values.** N. V. Chugreeva (Saratov Univ., USSR). *Zhur. Anal. Khim.*, 1960, **15** (4), 391–393.—The indicator properties of the 2, 4-dinitrophenylhydrazones of dihydroxytartaric acid, pyruvic acid and acetone, and of the *p*-nitrophenylosazone of dihydroxytartaric acid, were studied. It is shown that, in the temp. range 0° to 80°, these compounds can be used in soln. containing a high concn. of salts, ethanol or gelatin. Indicators of the hydrazone series can also be used for the photometric measurement of pH in the range 11·5 to 13·5, with an error of 0·1 to 0·2 per pH unit; Ca^{2+}, Sr^{2+}, Al^{3+}, Fe^{2+}, Fe^{3+}, Zn^{2+}, Mn^{2+}, Ag^{+} and Cd^{2+} can be present up to molar concn.; Mg^{2+} and Ni^{2+} interfere.

K. R. Cook

1348. **Characterisation of the colour quality of indicator transition. Complementary tristimulus colorimetry.** C. N. Reilley, H. A. Flaschka, S. Laurent and B. Laurent (Dept. Chem., Univ. of North Carolina, Chapel Hill, U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1218–1232.—The over-all quality of an indicator end-point is a composite function of the sharpness and colour quality of the transition. The former is related to the rate of reaction and the equilibrium constants and can be calculated rigorously, while the latter involves the colour phenomena in the soln. and their relation to the observing eye. By combining the principles of tristimulus colorimetry with Beer's law, formulae are developed to permit the complete characterisation of the colour quality factor, including a mathematical treatment of screening, dichromatism and the need for colour memory in many transitions. The theory permits the calculation of the relative quantities of inert dyes needed for screening a indicator; Kiton yellow 7GF (C.I. Acid Yellow 3 Acid red GA (C.I. Acid Red 1), FD & C Violet No. (C.I. Acid Violet 49) and Brilliant blue FCF (C. Acid Blue 9) are particularly suitable for th purpose. A. R. Rogers

1349. **The thermal stability of analytical standard** C. Duval and C. Wadier (Lab. de Rech. Microanal Paris). *Anal. Chim. Acta*, 1960, **23** (3), 257–26 (in French).—Decomposition curves obtained b means of the thermal balance are presented for 1 salts, *viz*, Ba acetate, Pb acetate, $Pb(NO_3)_2$, KNO $MgCl_2$, NH_4NO_3, ammonium citrate, $Co(NO_3)$ $UO_2(NO_3)_2$, Na terephthalate, Na_2S and K palm tate. Their i.r. spectra between 6 and 15 μ hav been examined, with soft paraffin as a binder an sodium chloride optics. It is concluded that P acetate, $MgCl_2$, $Co(NO_3)_2$ and Na_2S cannot b weighed directly for the preparation of standar soln. H. M.

1350. **Qualitative inorganic analysis. XVI. Us of a solution of hydrogen sulphide in acetone fo the precipitation of the copper - tin group of metals** W. I. Stephen (Dept. of Chem., Univ. Birmingham England). *Mikrochim. Acta*, 1960, (5–6), 927–93 (in English).—A saturated ($\simeq$ 2%) soln. of H_2 in acetone is stable for at least 3 months in th absence of H_2O or acid. For pptn. of the Cu - S group, after removal of alkaline-earth metals, sample vol. of 1·5 ml and an acid concn. of 0·35 are recommended. If As is absent, 5 or 6 drops o reagent are added, the soln. is warmed, and 2 o 3 drops more are added to ensure complete pptn If As is present, 4 drops of 10% NH_4I soln. ar added first. *Preparation of reagent*—Wash H_2 from a Kipp's generator with H_2O, dry with silic gel, and absorb in acetone (previously dried ove anhyd. Na_2SO_4) which is chilled in salt and ice Pass the gas until a saturated soln. is obtained.

P. D. Parr-Richard

1351. **Ion-exchange separation of groups o elements. III. Elements of the copper group** B. K. Preobrazhenskiĭ and Yu. P. Saĭkov. *Radio khimiya*, 1960, **2** (1), 68–72; *Ref. Zhur., Khim.* 1960, (18), Abstr. No. 73,013.—A scheme is pro posed for the qual. separation of elements of th copper group (Cu, Hg, Bi, Cd and Pb) by sorptio by the cationite KU–2.

IV. Elements of the third analytical group B. K. Preobrazhenskiĭ, V. P. Tsvelikhovskiĭ and V. I. Mel'nikov. *Ibid.*, 1960, **2** (1), 73–77.—Th method is based on the sorption from conc. HCl o the components (Mn, In, Ti, Hf, Zr, Co, Fe, Ga, U and Zn) by Dowex 1 (Cl^- form) and elution with various concn. of HCl. The elements not adsorbed (Cr, Be, V^{IV}, Tl^{I}, Ni, Al and the rare-earth metals) can be adsorbed by the cationite KU–2 (H^+ form) and eluted by various methods. C. D. Kopkin

1352. **Separation of cations by ion-exchange adsorption.** D. V. Bezuglyĭ, M. P. Borovshchuk and N. E. Khomenko. *Trudy Khar'kovsk. Politekh. Inst.*, 1959, **26**, 59–61; *Ref. Zhur., Khim.*, 1960 (18), Abstr. No. 73,014.—The method is based on the difference in the stability of the complexes o the cations with EDTA (disodium salt) (**I**), and has

en tested on synthetic mixtures prepared by ssolving freshly precipitated and washed $Fe(OH)_3$ **I** with subsequent addition of known amounts $CuSO_4$ and $MnSO_4$ and adjustment of the pH the soln. to 1·7 to 1·8. The soln. was passed rough a column containing 3 g of cationite U-1 (H^+ form), and the column washed and ıted with 0·5N-HCl (4 × 25 ml). The method ovides an efficient separation of micro amounts Cu and Mn from Fe. It was not possible to move Cu quant. from Zn with cationite KU-1 or ionite AN-2F, which is attributed to the small fference in the instability constants of the com- exes of these cations with **I**. C. D. KOPKIN

1353. **Chemical applications of complementary stimulus colorimetry.** C. N. Reilley and E. M. nith (Dept. of Chem., Univ. of North Carolina, apel Hill, U.S.A.). *Anal. Chem.*, 1960, **32** (10), 33–1240.—The principles of complementary tri- imulus colorimetry have been extended to the ıant. analysis of mixtures of two or three com- mponents with overlapping spectra, and the termination of dissociation constants of acids d the formulae and stability constants of complex ns. A simple analogue computer (described) is ed to calculate chromaticity co-ordinates within min. from data obtained by the weighted ordinate ethod at 10 wavelengths. A. R. ROGERS

1354. **Three-dimensional representation of oltammetric processes.** W. H. Reinmuth (Dept. Chem., Columbia Univ., New York, U.S.A.). *nal. Chem.*, 1960, **32** (11), 1509–1512.—Relation- ips established by time-dependent voltammetry e represented on a three-dimensional surface. tersections thereon may represent polarography, ronopotentiometry, polarography at a stationary ectrode, and constant-potential voltammetry. ood agreement is obtained with more rigorous eoretical treatments. The technique is applicable linear-current-scan chronopotentiometry and to herical electrodes. J. P. STERN

1355. **Chronopotentiometry with current reversal. pplication to *p*-benzoquinoneimine hydrolysis.** . C. Testa and W. H. Reinmuth (Dept. of Chem., olumbia Univ., New York, U.S.A.). *Anal. Chem.*, 960, **32** (11), 1512–1514.—By this technique it is ossible to prepare an unstable compound electro- emically and then to follow the course of its composition when the current is reversed. The chnique is illustrated by the anodic oxidation of aminophenol to *p*-benzoquinoneimine and subse- uent decomposition of the latter compound. The actions were carried out at constant current in rious concn. of H_2SO_4 and with a wide range of nes before current reversal. The rate constant of e decomposition could be deduced from the ratio the time to current reversal and the time to the st decomposition wave. G. BURGER

1356. **Chronopotentiometric potential - time rves and their interpretation.** W. H. Reinmuth ept. of Chem., Columbia Univ., New York, S.A.). *Anal. Chem.*, 1960, **32** (11), 1514–1517.— xpressions are derived for the potential - time lations for 14 reduction mechanisms. Criteria e presented by which these mechanisms can be stinguished. G. BURGER

1357. **Chemical kinetic parameters from chrono- potentiometric potential - time measurements.** A. C. Testa and W. H. Reinmuth (Dept. of Chem., Columbia Univ., New York, U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1518–1520.—The errors involved in the calculation of rate constants from chrono- potentiometric curves are discussed. The rate constant determined by Snead and Remick (*J. Amer. Chem. Soc.*, 1957, **79**, 6121) by potential - time measurement for the reduction of *p*-benzo- quinoneimine differs from that determined by the present authors (*Anal. Chem.*, 1960, **32**, 1512) by chronopotentiometry with current reversal. A method of calculation based on the fitting together of two curves, one derived from the error function and the other from the experimental results, is presented. The possible error due to the effect of the electrical double layer and the potential gradient in the soln. is considered and found to be negligible. G. BURGER

See also Abstracts—**1608**, Titration of EDTA and DCyTA. **1663**, Metallochromic indicator for Ca.

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

1358. **Flame-photometric methods in metallurgical analysis. A review.** J. A. Dean (Dept. of Chem., Univ. of Tennessee, Knoxville, U.S.A.). *Analyst*, 1960, **85**, 621–629.—The general possibilities of the application of flame photometry to the analysis of metallurgical products are discussed. Methods for determining constituents in various types of ferrous and non-ferrous alloys are reviewed under the heads of the elements to be determined. In many methods, solvent extraction serves to isolate the element in a form suitable for photometric deter- mination. The wavelengths, type of emission, and type of flame useful for the elements considered are tabulated, together with the emission sensi- tivities in p.p.m. (57 references.) A. O. JONES

1359. **Present state of methods for determining impurities in semi-conductor materials.** I. P. Alimarin and Yu. V. Yakovlev. *Zavod. Lab.*, 1960, **26** (8), 915–921.—A review with 48 references is presented. G. S. SMITH

1360. **The oxidation of some inorganic systems by lead tetra-acetate.** A. Berka, V. Dvořák, I. Němec and J. Zýka (Inst. for Anal. Chem., Charles' Univ., Prague). *Anal. Chim. Acta*, 1960, **23** (4), 380–384 (in French).—Soln. of Pb tetra-acetate free from Pb^{II} may be prepared by dissolving Pb_3O_4 in hot glacial acetic acid, crystallising out Pb tetra-acetate and re-dissolving it in acetic acid. The soln. may be used for the direct potentiometric titration of Cr^{2+}, Ti^{3+}, Fe^{2+}, Sn^{2+}, Sb^{3+} and As^{3+} in acid soln. (HCl or H_2SO_4 up to $\simeq$ 4N). T. R. ANDREW

1361. **Indirect volumetric determination of anions with redox indicators.** Z. Gregorowicz (Inst. anorg. Chem., Schlesische Techn. Hochschule, Gliwice, Poland). *Anal. Chim. Acta*, 1960, **23** (3), 299 (in German).—Sulphate, CrO_4^{2-} and PO_4^{3-} can be determined by reaction with an excess of 0·1N-$Pb(NO_3)_2$ in soln. buffered with acetic acid - Na acetate. The ppt. is filtered off and washed, and the excess of $Pb(NO_3)_2$ is titrated with 0·1N-$K_4Fe(CN)_6$ in the presence of 1 drop of 0·1N-$K_3Fe(CN)_6$ and 3 drops of 1% aq. Variamine blue acetate. The end-point is marked by a change in colour of the soln. from violet to milky yellow. The titration is carried out at $\simeq$ 60°. Bi- and ter-valent ions interfere, as do large amounts of Na, K, or ammonium salts. H. M.

1362. **Solvent extraction behaviour of some metal - 1-(2-pyridylazo)-2-naphthol chelates.** Shozo Shibata (Gov. Ind. Res. Inst., Nagoya, Japan). *Anal. Chim. Acta*, 1960, **23** (4), 367–369 (in English).—The extractability and colour of the extract is reported for the 1-(2-pyridylazo)-2-naphthol complexes of Ga, In, Cu, Bi, Ni, Zn, Fe, Hg, Mn, Co, Cd, Pb, Ce, Y and La for CCl_4, benzene, $CHCl_3$, ethyl ether and isoamyl alcohol. The absorption spectra and the effect of pH on extraction are reported for the complexes with Ni, Mn, Ga, In, Cd, Zn, Hg and Fe, together with the peak wavelengths for absorption and the molar extinction coefficients. T. R. Andrew

1363. **Ultra-violet spectra of some inorganic solids.** W. H. Waggoner and M. E. Chambers (Univ. Georgia, Athens, U.S.A.). *Talanta*, 1960, **5** (2), 121–126 (in English).—Ultra-violet spectra are presented for 9 sulphates, 3 chromates and 2 nitrates in aqueous soln. and in the solid state as KBr discs. The results on soln. were inconclusive except for the chromates, which gave maxima at 270 mμ and 370 mμ. With solid samples the sulphates gave maxima at 260 and 300 mμ, the chromates at 275 and 375 mμ and the nitrates at 280 to 290 mμ. The application of the technique to the qual. analysis of solid mixtures is described. W. T. Carter

1364. **Spectrophotometric titration of bivalent metals with 1-nitroso-2-naphthol in dimethylformamide.** I. T. Takahashi and R. J. Robinson (Dept. of Chem., Univ. of Washington, Seattle, U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1350–1352.—Small amounts (5 to 10 μmoles) of Cu and Ni may be titrated with a standard soln. of 1-nitroso-2-naphthol in dimethylformamide ($\simeq$ 0·005M) with photometric determination of the end-point. For Ca, best results are obtained at 600 mμ in 0·0015M-acetic acid in dimethylformamide ($\simeq$ 40 ml), while, for Ni, 525 mμ and a soln. containing 5 ml of butylamine and 35 ml of dimethylformamide are used. Results are reproducible to better than 1%. It was not possible to devise satisfactory conditions for the determination of Zn^{II}, Pb^{II} or UO_2^{II}. T. R. Andrew

1365. **Quantitative determination of the constituents of mixtures of non-corrosive light gases by the M.S.3. mass spectrometer.** U.K.A.E.A. (Production Group, Capenhurst). U.K.A.E.A. Report PG164 (CA), 1960. 13 pp.—Full experimental details are given of a procedure that is particularly suitable for the determination of H, He, O, N, Ar, CO and CO_2. The coefficient of variation is estimated to be 5% at the 1% level. G. J. Hunter

1366. **Use of ring-oven spot colorimetry with aid of a silver sulphide standard scale in inorga paper chromatography.** M. B. Ćelap and H. We (Chem. Inst., Belgrade, Yugoslavia). *Mikrochi Acta*, 1960, (5–6), 706–712 (in German).—T technique previously described (Weisz *et al.*, *Ib* 1959, 36) is applied to the semi-quant. determi tion of Pb, Cu and Cd, and Fe, Co and Zn, pre ously separated by paper chromatography, in t range 0·5 to 2 mg per ml with an accuracy wit ± 6%. J. P. Stern

1367. **Use of the ring oven in trace analys** G. Ackermann (Inst. f. Inorg. Chem., Bergaka Freiberg, Sachsen, Germany). *Mikrochim. Ac* 1960, (5–6), 771–774 (in German).—Trace impu ties in alkali- and alkaline-earth-metal salts a concentrated by pptn. as hydroxides or sulphic and collected on micro-filters (illustrated). The impurities are then examined by the ring-ov technique. The limit of sensitivity for the dete tion of Cu^{2+} and Fe^{3+} in 100 ml of N-KCl or N-acetate is 10^{-3} p.p.m. J. P. Stern

1368. **Separation of ions on filter-paper strips dissolution and precipitation zones.** E. Schwa Bergkampf (Chem. Inst., Montanist. Hochsc Leoben, Austria). *Mikrochim. Acta*, 1960, (5– 755–757 (in German).—In this extension of t ring-oven technique to paper strips, the strips a supported in a special device (illustrated) so th they may be warmed over a micro-flame. So of ions can be evaporated, leaving bands that c be further treated chemically, or fractiona dissolved and then pptd. on bands of reage deposited on the paper; Fe^{III}, Ni and Co (chlorides) can thus be identified by movement Ni and Co down the paper through bands diacetyl dioxime and 1-nitroso-2-naphthol (t coloured ppt. being readily identified), whilst t $Fe(OH)_3$ remains stationary and is identified wi $K_4Fe(CN)_6$. Difficulties encountered in the use this technique include decomposition of the pap under acidic conditions and the slow movement Cu in ammoniacal soln. J. P. Stern

1369. **Analysis of metal chelates by gas chrom tography.** W. J. Biermann and H. Gesser (Dep of Chem., Univ. of Manitoba, Winnipeg, Canad *Anal. Chem.*, 1960, **32** (11), 1525–1526.—Metals the form of acetylacetonato-complexes have be successfully separated on columns (4 ft. × 0·25 i of Apiezon L grease on glass beads 200 μ diameter. The complexes of Al and Be are separat from a soln. in acetylacetone and those of Al a Cr from a soln. in CCl_4. An argon ionisati detector is used. C. B. Baines

1370. **Extractive radiometric titration.** H. Spit (Paracelsus Inst., Bad Hall, Upper Austri *Mikrochim. Acta*, 1960, (5–6), 789–802 (in Germa —The method of Duncan and Thomas (*J. Ino Nuclear Chem.*, 1957, **4**, 376) is applied to t microgram range. A new measuring head, i corporating a stirrer and automatic discharge se is illustrated. The method is applied to the radi metric titration of Ag, Zn, Co, Cu and Hg wi dithizone with a non-isotopic or an isotopic ind cator. Determinations of 10 μg are possible wi high accuracy. J. P. Stern

1371. **X-ray fluorescence method for determin tion of the transition-metal content in very sm specimens of alloys.** P. J. Brown (Crystallograph

Lb., Cavendish Lab., Cambridge, England). *J. i. Instrum.*, 1960, **37** (10), 394–397.—The ratio the intensities of the characteristic X-rays nitted by two fluorescing elements in the specimen measured, and compared with the same ratio termined for a number of specimens of known mposition in the same physical state. When lly one fluorescing element is present a known nount of a second must be added. An accuracy tter than 1% is possible for specimens contain-g as little as 0·2 mg of the transition metals.

G. Skirrow

1372. **Spectrographic determination of metals in elding alloys.** E. S. Kudelya. *Avtomat. Svarka*, 960, (3), 76–79; *Ref. Zhur., Khim.*, 1960, (17), ostr. No. 69,246.—This is a modification of a ethod described previously [*Ref. Zhur., Khim.*, 55, (8), Abstr. No. 14,900]. High concn. of Cr, , Mn and W can be determined with an error of %. The comparison elements used are Fe and Ni.

K. R. Cook

1373. **Paper electrophoresis of mineral cations the presence of complexing agents.** P.-E. enger, I. Kapétanidis and W. von Janstein niv. de Genève, Switzerland). *Mikrochim. Acta*, 960, (5–6), 961–966 (in French).—The migration Ag, Hg^{2+} and Pb in the presence of 1,2-di--aminoethoxy)ethane-*NNN'N'*-tetra-acetic acid at fferent pH values is discussed and suitable buffer ln. from pH 2 to pH 12 are given. With Ag, nsiderable tailing occurs at low pH values; at H > 9, migration is anodic and a good spot is btained. At pH 2, Hg migrates to the anode and b to the cathode, but at pH 3 migration of Pb so becomes anodic. Separation of binary mixtures these ions should be possible.

P. D. Parr-Richard

1374. **Chelatometric determination of aluminium, ickel and manganese without prior separation.** . H. Wilkins (General Electric Res. Lab., Schenec-ady, N.Y., U.S.A.). *Anal. Chim. Acta*, 1960, **23** 4), 309–311 (in English).—To the sample soln. dd a known vol. (in excess of that required) f hydroxyethylethylenediaminetriacetic acid HEDTA) (**I**), adjust the pH to 5, add 10 ml of -Na acetate - M-acetic acid buffer, and boil, then ol the soln. to room temp. and add 3 drops of 1% soln. of Methyl Calcein or Methyl Calcein lue [condensation products of sarcosine (*N*-ethylglycine) and formaldehyde with fluorescein r with 4-methylumbelliferone, respectively] and ack-titrate with standard Cu^{II} soln. to determine l plus Ni. (The end-point is shown by a quenching f the fluorescence of the indicator.) Add NH_4F ·5 g), heat to boiling, and titrate the **I** liberated rom the aluminium complex with standard Cu^{II} oln. Add 1 g of K tartrate, adjust the pH to 9·5 ith NaOH, and titrate Mn^{II} with EDTA. Results n mixtures of ≃ 10 mg of each element show re-overies within ± 0·1 mg. T. R. Andrew

1375. **Automatic derivative spectrophotometric itration of excess EDTA in the determination of obalt, copper or iron.** H. V. Malmstadt and T. P. Hadjiioannou (Dept. of Chem. and Chem. Engng, niv. of Illinois, Urbana, U.S.A.). *Anal. Chim. cta*, 1960, **23** (3), 288–293 (in English).—To the ample soln. are added Calcein W (**I**), as indicator, nd a slight excess of EDTA (disodium salt) soln., hown by the appearance of a green fluorescence. his excess of EDTA is titrated automatically in he "Spectro-Electro" derivative titrator with standard $Cu(NO_3)_2$ soln., the **I** then functioning as an absorption indicator. An increase in absorbance at 500 mμ occurs when the excess of EDTA and the indicator have reacted completely with the titrant, and this is the signal for the automatic termination of the titration. The sample soln. is buffered to pH 4·4 with Na acetate - acetic acid, 4 drops of indicator soln. (0·1% of **I** in 0·001N-NaOH) are added and 0·04M-EDTA (disodium salt) is run in until a green fluorescence appears. The determination is then finished by automatic titration. Relative errors are within ± 0·1% when 5 to 50 mg of metal is taken. H. M.

1376. **Rapid deuterium determination in the presence of air.** P. H. Dutch (The Fluor Corp., Ltd., Whittier, Calif., U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1532.—Samples (5 to 10 ml) of hydrogen containing 30 to 70% of ^2H are analysed gas-chromatographically, with a thermal conductivity detector. With H as carrier gas, ^2H, O and N are readily distinguished on a 4-ft. column with molecular sieve 5A packing. The sensitivity for ^2H on a 5-ml sample is 500 p.p.m. J. P. Stern

1377. **Emission spectroscopic analysis of hydrogen - deuterium mixtures.** J. A. Parodi, W. G. Burch, L. F. Kendall and M. B. Leboeuf (Hanford Works, Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW–21718, July, 1951, Decl. May, 1960. 29 pp.—An emission-spectrometer procedure relating the intensities of Balmer lines to the relative concentrations of H and ^2H was calibrated for low flow rates. Isobars of true *vs.* apparent composition are given for the composition range of 89 to 100% of ^2H and for pressures of 0·7, 0·8, 0·9 and 1 torr. Experiments revealed that, with the preparation of similar calibration curves, the accurate analysis of H - ^3H mixtures could be carried out by the emission-spectrometer method. Nucl. Sci. Abstr.

1378. **Contribution to the analysis of alkali metals.** H. Koch (Inst. f. Angew. Radioaktivität, Leipzig). *Kernenergie*, 1960, **3**, 315–320 (in German).—By using light isotopes, the techniques of separating the alkali metals are checked. The chloroplatinic, perchlorate, and Kalignost (Na tetraphenylborate) methods do not give satisfactory analytical results. Also, in regions where apparently good values are obtained, satisfactory separations of the different alkali metals are not possible. This is true also for Na - Li separations with amyl alcohol, and ether - alcohol - HCl mixtures. None of these methods gives adequate results and they are not useful for separating mixtures which contain isotopes of alkali metals. By isotopic dilution analysis, Na can be determined in alkali-metal mixtures in a simple manner. An exact quant. determination of Na in complex materials is also possible, as is the determination of < 10% of sodium impurities in potassium hydroxide or potassium chloride.

Nucl. Sci. Abstr.

1379. **Polarographic determination of alkali metals in aqueous - alcoholic solution without supporting electrolyte.** G. M. Panchenkov, E. M. Kuznetsova and N. B. Akshinskaya (Lomonosov Univ., Moscow). *Zhur. Anal. Khim.*, 1960, **15** (4), 424–426.—The limits of useful concn. of alkali-metal chlorides were 0·0005N to 0·0070N in aq.

medium and 0·0020N to 0·0140N in aq. alcoholic medium (95% or 75% methanol, or 75% ethanol). A more negative value for $E_{\frac{1}{2}}$ was obtained in comparison with corresponding potentials observed in the presence of a supporting electrolyte. For example, the $E_{\frac{1}{2}}$ was − 3·82 V for Li^+, − 3·12 V for K^+, and − 3·08 V for Na^+, all in aq. soln., but a linear relationship between the height of the waves and the concn. was still obtained for the given concn. range. The most promising polarograms were obtained with a Li - K mixture in 95% methanol without a supporting electrolyte: two separate waves were shown. There is no separation of this mixture in aq. soln. K. R. Cook

1380. **Direct titration of potassium with tetraphenylborate. Amperometric equivalence-point detection.** D. L. Smith, D. R. Jamieson and P. J. Elving (Univ. of Michigan, Ann Arbor, U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1253–1257.—Tetraphenylborate ions are electrochemically oxidised at the graphite electrode. Potassium may be determined in soln. of pH 2 to 6 by titration with sodium tetraphenylborate soln. ($\simeq$ 3%) by means of a calomel - graphite electrode system at pH $\simeq$ 5 (acetate buffer), with an applied voltage of + 0·55 V on the graphite electrode. Results for known soln., $\not<$ 5 mM in K, are within 1% of the amount added. The major interferences with the method are NH_4^+, Rb^+, Cs^+, Tl^+, Ag^+, Hg^{2+} and nitrogen bases that react with sodium tetraphenylborate. Large amounts of SO_4^{2-}, Cl^-, PO_4^{3-} and acetate do not interfere. The method has been successfully applied to samples of limestone, glass and silica brick. T. R. Andrew

1381. **Flame-photometric determination of small amounts of rubidium in silicates.** E. A. Fabrikova (Inst. of Mineral., Geochem. and Crystallochem. of Rare Elements, Acad. Sci., Moscow). *Zhur. Anal. Khim.*, 1960, **15** (4), 427–430.—The effect of Cs, K, Na and Li on the intensity of rubidium emission (at 894·4 mμ) in the air - acetylene flame was studied, and it was found that the emission can be intensified and stabilised by the addition of K. Add conc. H_2SO_4 (0·4 ml), HF (15 ml) and HNO_3 (2 to 3 drops) to the mineral sample (0·5 to 1·0 g) and heat the mixture at 70° to 80° until H_2SO_4 fumes appear. Cool the product, add H_2O (2 ml) and conc. H_2SO_4 (0·1 ml) and again heat to fuming; evaporate for the third time with H_2O. Add H_2O (25 to 30 ml) to the residue, heat until no more will dissolve and dilute the soln. plus residue to 50 ml. Filter off the residue and reject the first portion of filtrate. Add K_2SO_4 soln. (25 mg of K per ml) (2·5 ml) to the filtrate (20 ml), dilute it to 25 ml, and measure the extinction at three different wavelengths—8 to 10 mμ above and below the rubidium line and on the rubidium line at 794·8 mμ. Simultaneously measure a standard soln. of rubidium sulphate with K_2SO_4 added at the same concn. The rubidium content is calculated from a given equation. This method allows of the determination of Rb in the parts per 10^5 and 10^6 range in a sample without a preliminary separation of Cs, K, Na, Li, Ca, Sr, Mg, P or Fe. The experimental error is 15%. K. R. Cook

1382. **Hexachlorotellurous acid as a precipitant for caesium.** H. A. C. Montgomery (Water Pollut. Res. Lab., Stevenage, Herts., England). *Analyst*, 1960, **85**, 687–688.—In the determination of radioactive caesium it is usually weighed as $CsClO_4$ or Cs_2PtCl_6 after separation from K and other el ments. Since the caesium and potassium salts hexachlorotellurous acid have widely differe solubilities in HCl, a rapid and convenient metho for pptn. of Cs with relatively slight co-pptn. of has been developed. Hexachlorotellurous aci reagent is added to the soln. of Cs in 11 to 12N-HC The reagent is prepared by dissolving 10 g TeO_2 in conc. HCl, evaporating the soln. to a sma bulk and diluting the cooled residue to 100 ml wit 11 to 12N-HCl. Since the ppt. of Cs_2TeCl_6 appreciably sol. in HCl, washing is kept to a min mum. The ppt. decomposes slowly in the presenc of moisture, especially when in contact with metallic surface, but samples on aluminium countin planchets may be protected for storage by moistei ing them with a 5% soln. of polystyrene in benzen and evaporating the solvent to give an imperviou film. A. O. Jones

1383. **Co-precipitation of caesium with thallium picrate.** I. M. Korenman, P. A. Ganichev an I. I. Melekhonova. *Trudy Khim. i Khim. Tekhnol* 1959, (1), 85–89; *Ref. Zhur., Khim.*, 1960, (18 Abstr. No. 72,970.—The isotope ^{134}Cs is used t study the co-pptn. of Cs^+ with Tl with an aq picric acid soln. The $TlNO_3$ soln. (0·6 ml, con taining 5 mg of Tl^+ and 40 μg of Cs^+) and 1% aq picric acid soln. (1 ml) were stirred in a centrifug tube for 30 min., and the ppt. was centrifuged of washed (× 2) with s-butyl alcohol and dissolve in 0·7 ml of HNO_3 (1:1); the co-pptd. Cs wa determined radiometrically. The influence on th co-pptn. of temp., time of standing of the ppt. i contact with the mother liquor, rate of mixing th reagents and other factors was studied. The rat of mixing the reagents and the time of standing c the ppt. have no effect on the co-pptn. of Cs. A increased temp., Tl picrate dissolves to a con siderable extent, causing a reduction in the tota amount of co-pptd. Cs from 58 to 29%; the amoun of Cs co-pptd. per mg of ppt. decreases from 2· to 2·2 μg. The addition to the sample of Cr^{3+} Fe^{2+}, starch, agar and gelatin does not affect th co-pptn. A small amount of Cs is co-pptd. wit freshly pptd. Tl picrate. It is concluded that th co-pptn. is isomorphous in nature. The pptn. o Tl with an aq. picric acid soln. does not separat Tl from Cs, because of the co-pptn. of Cs and th considerable solubility of the picrate. C. D. Kopkin

1384. **The use of picrolonic acid for the detectio of traces of copper with the electron microscope** E. Wiesenberger (Freie Univ., Berlin). *Mikrochim Acta*, 1960, (5–6), 946–960 (in German).—Electro deposition of Cu (0·005 to 0·00005 μg), followed b reaction with picrolonic acid, yields characteristi crystals which are clearly identified by the electro microscope. Full analytical details are given together with 17 electron micrographs. P. D. Parr-Richard

1385. **Lead diethyldithiocarbamate as a specifi reagent for copper.** I. Adamiec. *Rudy i Metal Nieżalazne*, 1960, **5** (10), 409–412 (in Czech).— Copper is detected in certain metals by dissolvin them in a suitable mineral acid, adding a CHCl soln. of Pb diethyldithiocarbamate and measurin the extinction of the organic layer. For Co, Zn and Al, HCl is used; for Pb, H_2SO_4; and for Pl with Ni, HNO_3 followed by H_2SO_4. The tim required for a determination is 20 min. and th limit of detection is 0·2 μg of Cu per ml of aci soln. P. Glover

1386. **Separation of copper as cuprous tetraenylborate.** D. G. Davis (Div. of Sci., Louisiana ate Univ., New Orleans, U.S.A.). *Anal. Chem.*, 60, **32** (10), 1321–1322.—*Procedure*—To 75 ml sample soln. (containing 2 to 60 mg of Cu and to 4 drops of conc. HCl) are added 10 ml of 1M-Na tetraphenylborate (**I**) and 10 ml of 0·1M-corbic acid (**II**). The mixture is set aside for 0·5 24 hr. and the ppt. is filtered off and washed th a soln. containing 10 ml of **I** and 10 ml of and 250 ml of water. The ppt. cannot be eighed as such but may be dissolved in HNO_3 d the copper determined by titration with 0·005M-DTA (disodium salt). Interferences by 11 metal ns are discussed. T. R. Andrew

1387. **o-(Toluene-*p*-sulphonamido)aniline (T-lphonamidine) as a new type of analytical agent. Gravimetric determination of cupric ion.** H. Billman, N. S. Janetos and R. Chernin (Dept. Chem., Indiana Univ., Bloomington, U.S.A.). *nal. Chem.*, 1960, **32** (10), 1342–1344.—In the urse of a study of sulphonamides as reagents it as found that *o*-(toluene-*p*-sulphonamido)aniline as a specific precipitant for Cu^{II}; pptn. is quant. the pH range 6·2 to 8·5. Interference is limited ions pptd. at this pH and to Ag^+, which is duced by the reagent. Most interferences are minated by working in tartrate soln.

T. R. Andrew

1388. **Determination of a small amount of copper titration with iodate.** I. L. Bagbanlÿ. *Trudÿ st. Khim., AN, Azerbaidzh. SSR*, 1959, **17**, 83–; *Ref. Zhur., Khim.*, 1960, (15), Abstr. No. ,029.—Add H_2SO_4 or HCl to a test soln. con-ining Cu^+ (0·08 to 3·8 mg), ensuring that the id concn. after the addition of the precipitating ent will be 2N, heat on a water bath, add an cess of 2·5% $NH_4[Cr(SCN)_4(NH_3)_2].H_2O$ soln. reshly prepared), set aside in a water bath until e supernatant liquid becomes clear, and then ter. Wash the ppt. with hot water until the rose nt is discharged, transfer the filter with the ppt. to the vessel in which the pptn. has been carried t, add hot 5% NaOH soln. (5 ml), and heat on water bath until the ppt. is completely decom-sed. Then add conc. HCl (at the end of the ration the soln. must be 4 to 4·5N with respect HCl), cool, add benzene (10 ml), and titrate th 0·005 to 0·1N-KIO_3 until the benzene layer is colorised; Fe^{2+} do not interfere with the deter-ination, but Fe^{3+} do, and may be masked by % KF soln. The method may be used for termining Cu in steel and cast iron.

K. R. Cook

1389. **Catalytic determination of microgram nounts of copper by amperometric measurements.** Czarnecki (Dept. of Inorg. Chem., Univ. Łódź, oland). *Chem. Anal., Warsaw*, 1960, **5** (3), 377–2.—Copper is a catalyst for the reaction $2Fe^{3+} + S_2O_3^{2-} \rightarrow 2Fe^{2+} + S_4O_6^{2-}$. Under specified nditions, the time of reaction is inversely pro-rtional to the concentration of Cu^{2+}. With o-electrode amperometry to follow the rate of action, 6 to 30 μg of Cu can be determined by easuring the half-times of reaction (*i.e.*, the time quired for the current to fall to one-half of its iginal value). The precision is claimed to be $\simeq$ %. The effects of temperature variation were ted, but not evaluated quantitatively.

P. Brych

1390. **Photometric determination of copper in the presence of nickel and cobalt.** L. B. Kristalëva and R. D. Tresnitskaya. *Ucheb. Zap. Tomskiĭ Univ.*, 1959, (29), 126–129; *Ref. Zhur., Khim.*, 1960, (16), Abstr. No. 65,068.—The method is based on preliminary separation of Ni and Co with dimethylglyoxime, followed by the photometric determination of Cu with Na diethyldithiocarbamate. K. R. Cook

1391. **Potentiometric investigation of several precipitation and complexing reactions for silver in strongly alkaline solution.** G. Facsko and R. Minges (Polytech. Inst., Timisoara, Romania). *Talanta*, 1960, **5** (2), 102–107 (in German).—Silver *p*-sulphamoylbenzoate can be dissolved in alkali and the Ag titrated potentiometrically with standard KCl, KBr, KI or KCN soln., with silver and satd. calomel electrodes and a KNO_3 salt bridge. The end-point is affected by the alkalinity of the soln.; to prevent this from changing during the titration, alkali must be added to the titrant. An error due to the oxidation of KI by dissolved O in the test soln. can be prevented by the addition of Na_2SO_3. The most accurate results are obtained with KCN. W. T. Carter

1392. **Determination of small amounts of silver in developed photographs.** N. N. Shishkina (Optical Inst., Leningrad). *Zhur. Anal. Khim.*, 1960, **15** (4), 431–436.—Place punched-out circles of film (diam. 5 mm) in quartz tubes and add 3N-HNO_3 to each tube (the amounts recommended for the dissolution of various possible silver contents are tabulated); the final HNO_3 concn. in the test soln. should be $\geq$ 0·1N. Heat the mixture, then dilute the cooled soln. and to an aliquot add 0·01% ethanolic *p*-dimethylaminobenzylidenerhodanine soln. (**I**) (0·4 ml of **I** per 5 ml of silver soln.). Stir the soln. without shaking and measure the extinction at 470 to 480 mμ. Take readings every 1·5 to 5 min. over a period of 10 to 20 min.; keep the soln. in the dark between readings, and refer the results to calibration curves. Keep the extinction values within the limits 0·08 and 0·2, which correspond to 0·1 to 1·0 μg of Ag per ml. This method permits of the determination of 0·04 μg of Ag per ml, *i.e.*, 5×10^{-5} g of Ag per 100 sq. cm of test layer. The accuracy is from 1 to 6% at concn. of 0·15 to 2·0 μg of Ag per ml. The reproducibility of the results is within 4%. K. R. Cook

1393. **New spot test for auric gold with *NN′*-di-(2-naphthyl)-*p*-phenylenediamine.** J. C. Soriano and E. Jungreis (Hebrew Univ., Jerusalem, Israel). *Talanta*, 1960, **5** (2), 127–128 (in English).—Tervalent gold gives a deep-red meriquinonoid compound with *NN′*-di-(2-naphthyl)-*p*-phenylenediamine at pH 3 to 8. The limit of identification is 0·1 μg of Au^{III} and the limit of dilution is 1 in 5×10^5; Fe^{III}, Ce^{IV} and Os^{VIII} interfere, but Fe^{III} can be masked with KF, and OsO_4 can be removed by boiling. Interference from $Fe(CN)_6^{3-}$ and SO_3^{2-} can be prevented by boiling with H_2O_2, that of MnO_4^- by reduction with As_2O_3 and Na_2CO_3, and that of S^{2-} by pptn. with $ZnCO_3$.

W. T. Carter

1394. **Spectrophotometric determination of beryllium with 2-phenoxyquinizarin-3,4′-disulphonic acid.** E. G. Owens, II, and J. H. Yoe (Pratt Trace Analysis Lab., Dept. of Chem., Univ. Virginia, Charlottesville, U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1345–1349.—At pH 6·0, 2-phenoxyquinizarin-3,4′-disulphonic acid reacts with Be to give a stable violet

complex (extinction $\simeq 0{\cdot}4$ at 550 mμ for 0·3 p.p.m. of Be in a 1-cm cell). The interference of most elements is eliminated by ion exchange, mercury cathode electrolysis, or addition of EDTA (calcium salt). Results are presented for the determination of Be in beryl, aluminium, copper alloys and steel. T. R. ANDREW

1395. **Spectrographic determination of traces of beryllium in ores by means of a sifter electrode.** J. Czakow and Z. Walewska (Inst. of Nuclear Res., Polish Acad. Sci., Warsaw). *Chem. Anal., Warsaw*, 1960, **5** (3), 369–375.—A semi-quant. extrapolation method, in which an internal standard is used, is described. The samples are held in a copper sifter-electrode, excitation is by a Feussner condensed spark and background correction is applied. The determinable range is 1 to 20 p.p.m. of Be and the coeff. of variation is ± 8·2%. P. BRYCH

1396. **The photo-neutron method for determining beryllium in ores.** F. I. Dvorechenskiĭ, I. P. Koshelev, B. M. Naĭdenov, S. N. Neuchesov, O. V. Shishakin and L. I. Shmonin. *Trudy Kazakhsk. Nauch.-Issled. Inst. Mineral. Syr'ya*, 1959, (1), 204–211; *Ref. Zhur., Khim.*, 1960, (18), Abstr. No. 73,047.—A method and apparatus are described for the photo-neutron determination of Be in ores by the reaction ^{9}Be (γ, *n*) ^{8}Be; ^{124}Sb (60 to 75 mC) is used as a γ-source, and BF_3 proportional counters connected with an adapted B–2 radiometer as neutron detector. The sensitivity of the method is 5×10^{-3} % of BeO with measurement for 10 min. on a sample of 50 to 180 g. The method has no upper sensitivity limit. The accuracy of the determination is independent of the presence of impurities (except B), moisture, and the degree of grinding of the sample. The influence of B is eliminated by means of a cadmium screen, but the sensitivity is then decreased by 25 to 30%. The method is simple and highly efficient. C. D. KOPKIN

1397. **Ion exchange and other chemical methods for beryllium-base alloys.** S. Kallmann, R. Liu and H. Oberthin (Ledoux and Co., Teaneck, N.J.). *U.S. Atomic Energy Comm.*, Rep. WADC–TR–59–325, May, 1959. 39 pp.—Procedures are described for both cation- and anion-exchange resins which enable all elements present in beryllium alloys to be determined in successive steps on one sample portion. The elements covered in the range of 0·1 to 10% are Cu, Al, Ni, Co, Ce, Ag, Au and Pd. NUCL. SCI. ABSTR.

1398. **Determination of fluorine in beryllium.** J. F. W. Smith and R. H. A. Crawley (Atomic Power Div., English Electric Co. Ltd., Whetstone, Middlesex, England). *Anal. Chim. Acta*, 1960, **23** (4), 345–350 (in English).—Fluoride is separated from Be^{2+} by steam-distillation from H_2SO_4 at 140° and subsequently determined photometrically in the distillate by the Zr^{IV} - sodium 4,5-dihydroxy-3-(4-sulphophenylazo)naphthalene-2,7-disulphonate (SPADNS) method of Bellack and Schouboe (*Anal. Abstr.*, 1959, **6**, 2794). T. R. ANDREW

1399. **Spectrographic determination of phosphorus in beryllium oxide.** I. A. Berezin and K. V. Aleksandrovich. *Zhur. Anal. Khim.*, 1960, **15** (4), 509–510.—Spectrograph ISP–22 is used with a slit width of 0·02 mm, a 14-amp. interrupted a.c. arc as light source, a burning time and interruption of 0·5 sec., and a total exposure time of 3 min. The standards containing 0·001 to 0·1% of P are pr pared by grinding calcium orthophosphate wit BeO. The sample is introduced into the ligh source on a moving cylindrical graphite electrod (length 80 mm, diam. 40 mm) rotating at 1 r.p.m the linear transporting velocity of the electrode 0·2 cm per sec. The upper electrode is a carbo rod (diam. 6 mm) and the electrode gap is 2 mm Type-III spectrographic plates are used. Th sensitivity of the determinations is 0·001% and th reproducibility of the results is ± 6%. K. R. COOK

1400. **Arsenate - iodimetric method of determ nation of magnesium in the presence of iron an aluminium.** G. B. Shakhtakhtinskiĭ and G. A Aslanov. *Azerbaidzh. Khim. Zhur.*, 1959, (4), 101 104; *Ref. Zhur., Khim.*, 1960, (16), Abstr. No 65,078.—In the method described, Al^{3+} and Fe^{3} (≯ 0·03N) are complexed with citric acid, Mg pptd. by Na_3AsO_4, and the AsO_4^{3-} in the ppt. a determined iodimetrically. K. R. COOK

1401. **Determination of magnesium an aluminium in zinc-based die-casting alloys.** I Bloch (H. J. van der Rijn, N. V. Res. Lab., Naarde Netherlands). *Anal. Chim. Acta*, 1960, **23** (3 233–239 (in English).—*Determination of Al*—Th alloy (turnings or drillings) (5 g) is dissolved in di HNO_3; Cu and Pb are removed by electrolysis, an the soln. is made up to 500 ml. An aliquot (100 m is treated with 20 ml of 50% aq. NH_4NO_3 sol and the soln. is neutralised to pH 3. The soln. cooled and poured, with stirring, into a beaker co taining 75 ml of water, 20 ml of 50% aq. NH_4NO soln. and 10 ml of aq. NH_3 (sp. gr. 0·91). This give a ppt. substantially free from Zn. The ppt. is filtere off, washed and dissolved in warm acid. Afte neutralisation to pH 3 the Al is re-pptd. as describe above, but with 8 ml instead of 10 ml of aq. NH The ppt. is filtered off, dried and ignited to constan weight. *Determination of Mg*—A similar sampl (10 g) is dissolved in dil. HCl; Cu, if present, dissolved by adding a few drops of HNO_3. The M is pptd. as $MgNH_4PO_4.6H_2O$ in the usual manne filtered and washed. The ppt. is dissolved in th minimum of warm 0·5N-HCl, and the soln. passed through an ion-exchange column (Dowe 1–X8) which is washed with water. The percolat and washings, now free from PO_4^{3-}, are evaporate to ≃ 25 ml, and buffered to pH 8 to 10. The M is titrated with 0·02N-EDTA (disodium salt), wit Eriochrome black T (**I**) or methylthymol blue (**I** as indicator. It is important to clean the titratio flask with boiling HCl (1 : 1) after each determina tion to remove residual traces of **I** which giv premature transient end-points. Soln. of **II** d not keep for more than a fortnight, and should b stored in a cool place in a dark bottle. A sma amount of Zn is co-pptd. with the Mg and shoul be allowed for in precise work. H. M.

1402. **Complexometric determination of thoriu and zirconium in magnesium alloys.** R. S Volodarskaya. *Zavod. Lab.*, 1960, **26** (8), 925 927.—*Determination of Th in the absence of Zr*— The sample (1 g) of magnesium alloy is dissolve in 30 ml of HCl (1 : 1), the soln. is diluted with 6 to 70 ml of water, aq. NH_3 is added until a lilac blue colour is obtained with Congo red pape 1 ml of 0·1% arsenazo soln. is added, and the sol is titrated with 0·025N-EDTA (disodium salt) to pink end-point. *Determination of Th in the presen*

Zr—The soln. of the alloy in HCl is treated with o 3 ml of 1% benzenearsonic acid soln. and boiled r 4 to 5 min. to precipitate the Zr. The cooled ln. is diluted to 200 ml and set aside until the t. has coagulated. A portion (100 ml) of the tered soln. is neutralised with aq. NH_3 and rated as described above. *Determination of Zr the absence of Th*—The sample (1 g) is dissolved 40 to 50 ml of HCl (1:1) and the soln., after lution with 20 to 25 ml of water, is treated with 025N-EDTA (disodium salt) so that there is an cess of 4 to 5 ml. The soln. is boiled for 2 to 3 in., then cooled to between 40° and 50°, and eated with aq. NH_3 until a lilac colour is obtained th Congo red paper. After addition of 2 ml of % sulphosalicylic acid soln. the excess of EDTA titrated with 0·025N-$FeCl_3$ to the colour change om yellow to pink. The method is satisfactory the presence of Ag, Cd, Zn, Al, Mn, Nd, La and . In the presence of Th, the Zr is pptd. from a % HCl soln. by benzenearsonic acid in an nount five times that of the Zr. The separated t. is made soluble by fusion. The EDTA soln. added in excess to a hot 20% HCl soln., which then boiled for 2 to 3 min. G. S. Smith

1403. **Photometric determination of calcium in agnesium alloys, with preliminary removal of agnesium by alkali in the presence of EDTA isodium salt).** L. Ya. Polyak. USSR Pat. o. 127,069 (10.3.1960).—With arsenazo (**I**) at H 12·5 to 13·0, calcium forms a soluble blue- olet complex; the sensitivity is 0·04 μg of Ca per l. The colour is developed instantaneously and stable for at least 3 days. The max. absorption at 570 mμ. The intensity is proportional to the ncn. of Ca in the range 0·01 to 0·1 mg per ml, nd the max. colour is developed at pH 12·0 to 3·0. In determining Ca in the presence of Mg, the g is pptd. with NaOH in the presence of EDTA isodium salt), the EDTA in the filtrate is oxidised y boiling with H_2O_2, and Ca is determined colori- etrically with **I**. No interference is caused by Al, n or residual Mg. The method can be used to etermine 0·01 to 2·0% of Ca in alloys.
C. D. Kopkin

1404. **Releasing effects in flame photometry. etermination of calcium.** J. I. Dinnin (U.S. eol. Survey, Washington, D.C.). *Anal. Chem.*, 960, **32** (11), 1475–1480.—Methods are described r overcoming the depressive effects on Ca, due to lphate, phosphate and aluminate, by the addition releasing cations; Sr^{2+}, La^{3+}, Nd^{3+}, Sm^{3+} and $^{3+}$ completely release the flame emission of Ca, nile Mg^{2+}, Be^{2+}, Ba^{2+} and Sc^{3+} release most of e Ca emission. The mechanism of the inter- rence and releasing effects, *i.e.*, the preferential rmation of certain compounds in the flame, is plained on the basis of chemical equilibria in the oplets of soln. evaporating in the flame. The sults presented show a greater flexibility in the oice of releasing agent than was previously ssible. A releasing agent may be selected on the sis of molar effectiveness, background emission, availability and cost of reagent. The method is pplied to the determination of Ca in silicate rocks thout using separation techniques, and results e in good agreement with those of a chemical ethod of analysis. C. B. Baines

1405. **Determination of traces of nitrogen in etallic calcium.** C. Den Tandt (Inst. Inter-univ. ss Sciences Nucléaires, Brussels). *Mém. Sci. Rev. étall.*, 1960, **57** (9), 676–682.—Calcium purified by vacuum sublimation is very reactive; an apparatus is described in which a soln. of the sample is safely prepared, with cooling, in an inert atmosphere. *Procedure*—After introduction of calcium (10 g) purge the apparatus with a rapid stream of dry Ar. Reduce the flow of gas and add the following soln. in the order given—a mixture (1:1) of HCl (1:3) and twice-distilled glycerol (5 ml), HCl (1:3) (15 ml) and sufficient HCl (2:1) to complete dissolution of the sample. Dilute to 250 ml and transfer 25 ml to a semi-micro Kjeldahl apparatus. Add 15 ml of 40% NaOH soln. and steam-distil, trapping the NH_3 in 20 ml of 0·02N-HCl in a 100-ml flask. When the receiver is three-quarters full add 5 ml of Nessler reagent, dilute to 100 ml and after 10 min. measure the extinction at 450 mμ. Compare with a standard curve. The method is valid for up to 200 μg of N per 100 ml, with a reproducibility of $\pm$ 4 μg.
A. J. Bennett

1406. **Photometric determination of strontium.** A. S. Karnaukhov, M. Mizera and R. Palaush (Higher Pedagog. School, Prague). *Zhur. Anal. Khim.*, 1960, **15** (4), 502.—*Procedure*—Set aside a mixture of test soln. (< 1·75 mg of Sr per ml) (5 ml) and 0·05N-dilituric acid (**I**) (5 ml) for 15 min. With higher concn. of Sr use more **I**. Filter the mixture and measure the extinction of the filtrate at 420 mμ, in a 0·5-cm cell. Construct a calibration curve. With low concn. of Sr (up to 2 mg per ml) the max. error is 2%, but this increases with increase in concn.; certain cations (NH_4^+, K, Sn, Fe, Cr, Cd, Cu, Ca, Ba, Zn, Co, Ni, Hg, Ag and Pb) interfere. K. R. Cook

1407. **Flame-photometric determination of barium and sulphate: an improved technique.** D. C. Cullum and D. B. Thomas (Colgate-Palmolive Ltd., 371 Ordsall Lane, Manchester, England). *Analyst*, 1960, **85**, 688–689.—In a method previously reported (*Ibid.*, 1959, **84**, 113) for the determination of SO_4^{2-} in detergents, the SO_4^{2-} were pptd. as $BaSO_4$, which was separated by centrifuging and suspended in 1% starch soln. for analysis by flame photometry. In this modification the $BaSO_4$ is dissolved in EDTA soln. (ammonium salt). The sample ($\equiv$ 0·1 to 0·2 mg of SO_4^{2-}) in aq. soln. is treated with conc. HCl and diluted to 100 ml. A 5-ml portion is treated with 5 ml of 1% $BaCl_2$ soln. and the ppt., after being washed by centrifuging, is dissolved by addition of the EDTA soln. to exactly 10 ml. A 5-ml portion of the standard soln. of H_2SO_4 or Na_2SO_4 (2000 p.p.m.) is treated identically. The E.E.L. flame photometer is set to zero with the EDTA soln., and to full deflection with the soln. containing 1000 p.p.m. of SO_4^{2-}, and the sample is then examined. The SO_4^{2-} (%) is the scale reading divided by five times the sample wt. The sample must be freed from surface-active matter by the method previously described; anions other than SO_4^{2-} that form insol. barium salts must be absent. A. O. Jones

1408. **Determination of barium in minerals.** A. de Sousa (Fac. of Sci., Univ. Lisbon, Portugal). *Chemist Analyst*, 1960, **49** (3), 75–76.—After dissolution of the sample, the HCl soln. (containing 0·15 to 0·3 g of Ba) is treated with $K_2Cr_2O_7$ and the $BaCrO_4$ is collected by filtration; an EDTA titration completes the determination. *Procedure*—Dissolve the ppt. of $BaCrO_4$ in 100 ml of HCl (1:1) containing 2 g of KI and dilute to 1 litre. Transfer a 500-ml aliquot to each of two beakers and precipitate the Cr with aq. NH_3. Filter, wash, and

evaporate the filtrates and washings to 200 ml. To one beaker add 200 ml of ethanol, 8 to 12 drops of 0·1% metalphthalein soln. (phthalein complexone) (Anderegg *et al.*, *Anal. Abstr.*, 1954, **1**, 1180) and 2 ml of conc. aq. NH_3, and titrate with 0·1M-EDTA (disodium salt) until colourless. If pptn. occurs and obscures the end-point, titrate the soln. in the second beaker, but add the ethanol just before the end-point. Results are slightly higher than those obtained gravimetrically.

P. D. Parr-Richard

1409. **Simultaneous determination of barium and strontium in silicate rocks, by the method of addition, by emission spectroscopy in an electric arc.** V. Gabis (Lab. de Min.-Crist., Fac. des Sci., Paris). *Compt. Rend.*, 1960, **251** (2), 232–234.—The technique of successive additions has been used for samples in which the Ba and Sr were in the form of chlorides, sulphates and calcined oxides. The most reproducible results were obtained when the elements were in the form of sulphates and the addition was made by impregnation of the graphite. A d.c. arc was used with c.d. 4·5 amp. for clays after attack by HF - HCl or HF - H_2SO_4 and 7·5 amp. for calcined clays. The relative intensity of the lines due to Sr (4607·33 A) and Ba (4554·04 A) varies linearly with the concn. of these added metals provided that the concn. of Ba is $\gtrsim 150 \times 10^{-6}$. The lines Mg (5183·62 A) or K (5782·60 A) (for additions as chloride diluted with KCl) are used as internal reference lines. J. H. Waton

1410. **Micro-determination of zinc by chromatography and colorimetry.** F. Burriel-Martí and R. Gallego Andreu (Dept. Chim. Anal., C.S.I.C., Madrid, Spain). *Mikrochim. Acta*, 1960, (5–6), 912–916 (in French).—A chromatographic separation of 5 μg of Zn from Cu (150 μg), Fe (150 μg), Mn (1 μg), Co (25 μg), Ti (25 μg), Ni (25 μg), Cr and Pb is described. By using Whatman No. 1 paper (purified by washing with 1% aq. EDTA and water) and butanol - 2N-HCl (1:1) as solvent, an R_F value of 0·75 is obtained for Zn; values for the other metals are, respectively, Cu 0·24, Fe 0·20, Mn 0·10, Co 0·08, Ti 0·00, Ni 0·05, Cr 0·00, and Pb 0·00 (some tailing). The spot due to Zn is located with dithizone soln. (0·01% in CCl_4) (sensitivity 1 μg of Zn) and the Zn dithizonate is exhaustively extracted with 2N-HCl before the colorimetric determination with dithizone.

P. D. Parr-Richard

1411. **Spot-test detection of cadmium in plating.** J. G. Sabo (54 Bergen Avenue, Clifton, N.J., U.S.A.). *Chemist Analyst*, 1960, **49** (3), 78.—The test surface is treated with 1 to 2 drops of satd. aq. NH_4NO_3 soln. and, after exactly 45 sec., the liquid is adsorbed on to filter-paper. Addition of 1 drop of 10% Na_2S soln. to the spot gives a yellow stain in the presence of Cd. Zinc, Mn and Pb are only slightly attacked and Be, Ti, Zr, Cr, Mo, Fe, Co, Ni, Cu, Ag, Hg, Al, Sn, Sb and Bi are not attacked within 45 sec. P. D. Parr-Richard

1412. **New rapid gravimetric method for the determination of cadmium.** I. Grecu (Fac. Pharm., I.M.F., Cluj, Romania). *Stud. Cercet. Chim., Cluj*, 1959, **10** (2), 295–298.—The proposed method is based on the formation of a complex of Cd^{2+} with piperazine in the presence of excess of NH_4SCN. *Procedure*—The sample ($\simeq$ 10 to 30 ml) is treated with 2 g of NH_4SCN, then acidified with 2 or 3 drops of acetic acid, and 25 ml of a 4% soln. of piperazine is added in the cold. The pptd. $[Cd(C_4H_{10}N_2)](SCN)_2$ is filtered off and washe successively with a soln. containing 0·2% each piperazine and NH_4SCN, a soln. containing 0·04 of each, 1% piperazine soln. in ethanol - eth (1:3) (5 × 2 ml), and ether containing 0·1% piperazine (4 × 2 ml), and vacuum-dried. Th coeff. of variation is ± 0·25%, and the sensitivi is 80 μg per ml. Alkali and alkaline-earth metal Hg^{2+} and Mn^{2+} do not interfere, but Cu^{2+}, Zn^{2} Co^{2+}, Ni^{2+}, Pb^{2+}, Al^{3+} and Cr^{3+} do. H. Sher

1413. **Co-precipitation of small amounts cadmium with certain metal hydroxides.** V. Plotnikov. *Zhur. Neorg. Khim.*, 1959, **4** (12), 2775 2778; *Ref. Zhur., Khim.*, 1960, (12), Abstr. N 46,970.—The radioactive isotope ^{115}Cd is used t study the co-pptn. of small amounts of Cd wit the hydroxides of Be, Al, Cr, Ti, Fe, Bi and Pb a a function of the concn. of aq. NH_3, the amount hydroxide pptd., the temperature, the time standing of the ppt., and other factors. The c pptn. of Cd with $Fe(OH)_3$ decreases with increas of the NH_4^+ concn. On increasing the Cd conte of the soln., the percentage co-pptn. decrease but the absolute amount of Cd in the ppt. is i creased. The amount of co-pptd. Cd also increase with increase of the amount of $Fe(OH)_3$; at p 6·9 to 9·7, in the absence of ammonium salt 4 μg of Cd is almost completely co-pptd. with 7 m of Fe^{3+}. Increasing the time of standing of th ppt. from 30 min. to 30 hr. causes a slight decreas in the co-pptn. only in alkaline medium. Increas of temp. decreases the co-pptn. The co-ppt increases in the order Pb^{2+}, Be^{2+}, Bi^{3+}, Al^{3+}, Cr^{3} Fe^{3+} and Ti^{4+}. There is practically no co-ppt with $Pb(OH)_2$. The maximum co-pptn. is at p 7 to 8, which corresponds to complete pptn. $Cd(OH)_2$ when aq. NH_3 is added to pure soln. Cd salts. It is shown that the co-pptn. of Cd wit the hydroxides of the metals named is due t adsorption. C. D. Kopkin

1414. **Application of the molten zinc electrod to the spectrographic determination of small amount of cadmium in zinc.** B. Czerny-Hołyńska (Dept of Metallurgy of Non-Ferrous Metals, Acad. o Mining and Metallurgy, Cracow, Poland). *Chem Anal., Warsaw*, 1960, **5** (3), 383–388.—By usin a molten zinc electrode similar to that of Frederick son and Churchill (*Anal. Abstr.*, 1954, **1**, 2063 the effects of non-homogeneity of samples wer nullified, and as little as 0·004% of Cd could b determined, as compared with 0·05% with soli electrodes. The best results were obtained a 450°; zinc vapours interfere at higher temp.

P. Brych

1415. **Detection of mercuric ions with potassiun antimonyl tartrate.** E. Chinoporos (Suffolk Univ Boston, Mass., U.S.A.). *Anal. Chem.*, 1960, **3** (10), 1364.—When a soln. of potassium antimony tartrate (4%) (2 ml) is added to a soln. containin Hg^{II} [0·1% as $Hg(NO_3)_2$] (3 ml) and the mixture i made alkaline with aq. NH_3, a grey - black ppt. i produced. This is not formed by 18 other commo ions examined and the reaction is suggested as a qualitative test for Hg^{II}. T. R. Andrew

1416. **Volumetric determination of mercurou ions with alkali-metal ferrocyanide solutions wit bromophenol blue as adsorption indicator.** H Basińska and A. Bukowska (Dept. of Inorg. Chem Copernicus Univ., Toruń, Poland). *Chem. Anal. Warsaw*, 1960, **5** (3), 355–360.—Mercurous ions ca

titrated with K, Na or Li ferrocyanide in acid n. (0·1N-HNO_3). Bromophenol blue as adsorp- n indicator gives a good colour change from rple - pink to grey - green. Cations forming ol. ppt. (*e.g.*, Zn, Pb and Cu) interfere. The curacy on 25 to 250 mg of Hg is within 0·8% titrations). P. Brych

1417. **Titration of mercury and nickel salts with steine solutions.** M. Wroński (Dept. of Chem. chnol., Univ., Łódź, Poland). *Chem. Anal., arsaw*, 1960, **5** (3), 511–512.—Methods are scribed for the determination of (*i*) cysteine th $Hg(NO_3)_2$ or Na *o*-hydroxymercuribenzoate and (*ii*) Hg^{2+} or Ni with cysteine. *Procedure* (*i*)—To a soln. of cysteine (1 to 70 mg) add ml of N-NaOH and 0·2 ml of 0·1% ethanolic thizone, dilute to 100 ml and titrate with 005N to 0·05N-$Hg(NO_3)_2$ or **I** to a red end-point. he accuracy is 0·2 to 1%. *Procedure for* (*ii*)— 100 ml of a soln. containing < 50 mg of Hg^{2+} d 5 ml of N-aq. NH_3, clear with 5 ml of 3% $_2SO_3$ soln., add 1 ml of thiofluorescein soln. 1% in 0·2N-aq. NH_3) and titrate with cysteine ln. (0·03N in 0·01N-H_2SO_4) to a permanent blue d-point. Nickel titrations are carried out nilarly, except that the indicator is decolorised th **I** and an indicator correction applied. The curacy is 0·2%. P. Brych

1418. **Separation and spectrophotometric determination of microgram quantities of mercury using ethyldithiocarbamate.** E. A. Hakkila and G. R. Waterbury (Univ. California, Los Alamos Sci. Lab., Mex., U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1340–1342.—Mercuric ions are extracted with ethyldithiocarbamate (sodium salt) and CCl_4 from ln. containing EDTA (disodium salt) and KCN pH 9·3 to 10·0. The extinction of the extract is easured at 278 mμ. Of 32 ions studied only Cu, III and Bi^{III} interfere seriously, but Ru should removed by hypochlorite oxidation and volatilis- ion if more than 200 μg is present. The method suitable for 1 to 50 μg of Hg. Coeff. of variation 5% and 2% (14 determinations) are reported r 10 and 50 μg of Hg, respectively.

T. R. Andrew

1419. **Alkalimetric titration of boron after mercury-thode and hydroxide separations of interferences.** C. Neace (Nuclear Div., The Martin Co., Balti- ore, Md., U.S.A.). *Chemist Analyst*, 1960, **49**(3), 70–72.—Interfering elements (*e.g.*, Al and U) maining in the soln. after mercury-cathode paration are removed by hydroxide pptn., and rate is determined in the filtrate. *Procedure*— fter mercury-cathode separation, transfer the ln. to a polyethylene beaker and add 50% NaOH ln. to pH 7·5. Filter, acidify the filtrate with 2 ops of H_2SO_4 (1:4) and heat to expel CO_2. Cool, our into a polyethylene beaker and adjust the pH 7·3. Add mannitol (7 g) and titrate with 0·005N- aOH to restore the pH to 7·3. The accuracy is thin 0·002% for > 0·1% of B.

P. D. Parr-Richard

1420. **Photometric determination of boron by lvent extraction using monomethylthionine.** L. C. asztor and J. D. Bode (Jones & Laughlin Steel orp., Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 960, **32** (11), 1530–1531.—*N*-Methylthionine (Azure is superior to methylene blue in Ducret's method r determining B (*Anal. Abstr.*, 1958, **5**, 1793) ecause it gives a lower reagent blank and permits a wider range (0·1 to 200 μg of B) to be determined. The extinction of the extracted complex at 658 mμ is a measure of the B content.

J. P. Stern

1421. **Determination of microgram amounts of boron in complex alloys.** R. B. Golubtsova (Baĭkov Inst. of Metall., Moscow). *Zhur. Anal. Khim.*, 1960, **15** (4), 481–486.—*Procedure for nickel, cobalt and iron alloys*—With gentle heating dissolve the sample (0·05 g), consisting of finely ground turnings, in H_2SO_4 (1:2) (1 ml), cool, add 25% $(NH_4)_2S_2O_8$ soln. (0·5 ml) in the presence of 1% $AgNO_3$ soln. (0·2 ml) and boil the mixture under reflux for 15 min. Filter off the ppt. (W and Nb), wash with H_2O, dilute the filtrate to 15 ml and transfer it to a water-cooled electrolytic cell with a mercury cathode; cool to > 20°. After electrolysis for 15 to 20 min., test the colourless soln. for the absence of Ni, Co, Fe or Cr (depending on the alloy). Run off the soln. from the cell, wash the mercury surface with H_2O (4 × 2·5 ml), add 40% NaOH soln. (2·5 ml) to the soln. and heat it on a water bath to coagulate the ppt. Filter off the ppt. (Ti and Zr), wash it with 1% NaOH soln. (3 × 1 ml) and discard it. Add conc. H_2SO_4 dropwise to the filtrate till the soln. is acid to Congo red paper, then add more conc. H_2SO_4 (10 ml) and HNO_3 (1:40) (2 to 3 drops) and evaporate the soln. twice to fuming (≃ 5 min.). Cool, add 0·005% carminic acid (**I**) soln. (10 ml) in conc. H_2SO_4 and set the mixture aside for at least 4 hr. [Chromotrope-2B (**II**) (C.I. Acid Red 176) or arsenazo II (**III**) may also be used as the reagent.] Measure the extinction at 575 mμ in a 10-mm cell against a blank. Calibration curves should be constructed from soln. of B-free alloys to which B has been added as a standard H_3BO_3 soln. *For titanium alloys*—Dissolve the sample (0·01 g) in H_2SO_4 (1:3) (1 ml) as described above, add 15% $(NH_4)_2S_2O_8$ soln. (0·15 ml), boil the mixture as before, and then cool it; the soln. should be clear. Add 40% NaOH soln. (2·5 ml), and heat the soln. on a water bath to coagulate the ppt. Filter off and wash the ppt. with 1% NaOH soln. (3 × 1 ml). If Cr or Ni is present electrolyse the filtrate with a mercury cathode. To the filtrate add conc. H_2SO_4 (7 ml) and conc. HNO_3 (5 drops), and evaporate twice until white fumes appear (≃ 5 min.). Cool the soln. add 0·025% **I** soln. (2 ml) or 0·01% **II** soln. (3 ml), or 0·005% **III** soln. (3 ml), and measure the extinction of the soln. in a 10-mm cell at 575 mμ (with **I**), 620 mμ (with **II**) or 640 mμ (with **III**). These methods can be used to determine 0·01% or more of B in alloys.

K. R. Cook

1422. **Emission spectrographic method for determination of minute traces of boron in reactor graphite.** M. Brandenstein, I. Janda and E. Schroll. *Mikrochim. Acta*, 1960, (5–6), 935–945 (in German).—The sensitivity for the spectrographic determination of B in graphite was increased from 0·01 p.p.m. to 0·003 p.p.m., or higher, by partial ashing, use of a double arc, and volatilisation of thermostable B_4C by addition of fluoride mixtures; NaF - AlF_3 or LiF - AlF_3 was most effective. Arsenic was used as the reference material.

P. D. Parr-Richard

1423. **Determination of boron and borides of some metals.** A. T. Pilipenko and L. N. Kugaĭ. *Ukr. Khim. Zhur.*, 1959, **25** (6), 786–788; *Ref. Zhur., Khim.*, 1960, (15), Abstr. No. 61,079.—Decompose the borides of Ti, Zr, Nb, Ta, Cr and

W by alkali fusion in iron crucibles at 300° to 400°, then add Na_2O_2 and fuse again. Dissolve the melt with H_2O, neutralise to methyl red, precipitate the metals with a $BaCO_3$ suspension, and determine B in the filtrate by alkaline titration in the presence of invert sugar or mannitol. Decompose nickel boride with HNO_3, plus a few drops of HCl, by boiling under reflux, neutralise, precipitate Ni with Na diethyldithiocarbamate, extract with $CHCl_3$, acidify the aq. layer and boil under reflux to decompose diethyldithiocarbamic acid; determine B as described above. Decompose the borides of Mo and V by alkali fusion in nickel crucibles as described above; separate Mo, V and Ni (from the crucible) by the extraction of the diethyldithiocarbamates and determine B as described above.

K. R. Cook

1424. **Infra-red spectrophotometric determination of diborane(6) [B_2H_6], dichloroborane and trichloroborane in mixtures.** H. G. Nadeau and D. M. Oaks, jun. (Olin Mathieson Chem. Corp., New Haven, Conn., U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1480–1484.—The method described is especially useful since it is rapid and relatively more sensitive than the wet chemical analysis because of the strong absorption in the infra-red region shown by boron compounds. The individual components are determined in mixtures directly from their characteristic absorption peaks—diborane(6) at 5·35 μ, dichloroborane at 8·98 μ and trichloroborane at 10·24 μ. All three components have mutual absorption bands in the regions most likely to be used for analysis and so this method is applied by using Vierordt's method (*cf.* Gillam *et al.*, "Introduction to Electronic Absorption Spectroscopy in Organic Chemistry," pp. 185–186), with simultaneous equations to correct for overlapping absorption. The procedure has shown an over-all precision on synthetic samples of $\pm$ 2% of the three compounds in the concn. ranges studied. General interference with the analysis can be expected in the presence of compounds containing B–H and/or B–Cl linkages.

C. B. Baines

1425. **Determination of decaborane with dipyridylethylene.** E. A. Pfitzer and J. M. Seals (Univ., Pittsburgh, Pa., U.S.A.). *Amer. Ind. Hyg. Ass. J.*, 1959, **20**, 392–395.—A new reagent, 1,2-di-(4-pyridyl)ethylene (**I**), is proposed for the detection of $B_{10}H_{14}$ in xylene or in air. A 0·5% soln. in xylene was found to be best suited for the detection. **I** added to a soln. of $B_{10}H_{14}$ in xylene gives a clear pink to red soln. with an absorption peak at 515 mμ. At room temp. the colour reaches a max. intensity within 30 min., is stable for at least 1 hr., and fades slowly. The principal advantages of this reagent over quinoline (*cf.* Hill and Johnston, *Anal. Abstr.*, 1955, **2**, 3311) are increased sensitivity, more rapid colour development, and lower reagent-blank readings. Limited experiments indicate that the reagent in a gas-bubbler collecting-bottle absorbs $B_{10}H_{14}$ from current of air.

Chem. Abstr.

1426. **Use of neutron activation analysis for determining effectiveness of zone-refining techniques in the purification of aluminium.** W. D. Mackintosh (Atomic Energy of Canada, Ltd., Chalk River, Ontario). *Anal. Chem.*, 1960, **32** (10), 1272–1275.—Thirteen impurity elements in a bar of zone-refined aluminium were determined by neutron activation analysis and the results were used to indicate the degree to which the refining reduced the concn. of these impurities in central region of the bar. K. A. Procto

1427. **Chronopotentiometric study of galli** E. D. Moorhead and N. H. Furman (Frick Ch Lab., Univ., Princeton, N.J., U.S.A.). *A Chem.*, 1960, **32** (11), 1507–1509.—Voltammetry const. current with a mercury-pool electrode sh that in 0·1M-KNO_3 the quarter-wave potent ($E_{0·25}$) for 10^{-3}M-$Ga(NO_3)_3$ are — 1·02 and — 0·7 *vs.* the S.C.E. for the cathodic and anodic wa respectively. The reaction is affected by ox film formation. In 7·5N-KSCN, the $E_{0·25}$ for $\times$ 10^{-3}M-$Ga(NO_3)_3$ is — 0·90 V for each wave good agreement with polarographic values. cathodic transition time is nearly three times anodic transition time. The Ga(Hg) - Ga^{III} cou in 7·5 to 10N-KSCN is reversible, and the precis measurable anodic transition time is suitable analytical application. J. P. Stern

1428. **Radiochemical evaluation of solvent extr tion of indium as the bromide at high concentratio** E. B. Owens (Lincoln Lab., M.I.T., Lexingt Mass., U.S.A.). *Anal. Chem.*, 1960, **32** (10), 13 1367.—Indium in concn. up to 0·6M can be extr ted from soln. in HBr with pentan-2-one, leav only trace quantities (6×10^{-5}M) of indium a much of the impurities in the acid layer.

K. A. Procto

1429. **The chemistry of thallium and arse Bromatometric determination of thallium in prese of arsenic.** E. Schulek and L. Pataki (Inst. Inorg. and Anal. Chem., L. Eötvös Univ., Bu pest, Hungary). *Mikrochim. Acta*, 1960, (5–6), 7 720 (in German).—Boiling H_2SO_4 quant. redu Tl^{III} to Tl^{I} in 60 min.; both As^{III} and As^{V} unaffected; Tl (10 mg) can thus be determined titration with $KBrO_3$ in the presence of 500 mg As_2O_3, with an accuracy within $\pm$ 0·35%. Copp Fe, Sn, Mg and Ag do not interfere, but V^{IV} mal recognition of the end-point difficult. *Procedure* Warm the sample (10 ml, containing 10 mg of T dilute with water (100 ml) and boil with co H_2SO_4 (5 ml) for 5 min. Add 20% HCl (1 ml) a KBr (0·2 g) and titrate with 0·01 or 0·1N-KBr with *p*-ethoxychrysoidine as indicator, to a pa yellow end-point. J. P. Stern

1430. **Evaluation of flame photometry for determination of elements of the rare-earth gro** O. Menis (Oak Ridge National Lab., Tenn., U.S.A *U.S. Atomic Energy Comm.*, Rep. CF–52–81 1959. 20 pp.—Flame spectra are presented of rare-earth elements, except Ce and Pm, and of closely related elements, Sc and Y. The prec wavelength and relative spectral intensity of ea line and band, as well as the width of all ban are tabulated. In addition, the major ban and lines of each element are listed separate together with the half-band width of all ban Also included in this tabulation are the relati spectral interferences of other elements of the gro studied which interfere with the emissivity measu ments of the bands and lines listed. The wa lengths of band crests and lines were fixed fr spectral data recorded photographically with prism spectrograph. The remaining spectral da are based on the several elements which w recorded with a high-sensitivity grating fla spectrophotometer. Operating conditions used recording the spectra with the spectrograph a

• with the flame spectrophotometer are given. ed on the spectra and spectral data presented, uctions can be made relative to the applicability . limitations of flame photometry in the deter- nation of Sc, Y and rare-earth elements in ttures thereof. Lanthanum, Y and Nd can be ermined without spectral interference by other ments of the group studied. For all other ments of the group, some spectral interference is ountered. Nevertheless, by judicious selection wavelength, certain of the elements may be ermined flame-photometrically in the presence nany of the other elements of the group without ous interference. Two examples are cited.

Nucl. Sci. Abstr.

1431. **Anion effect in the flame-photometric ermination of the rare-earth elements.** N. S. uéktov and L. A. Ovchar (Inst. of Gen. and rg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 60, **26** (8), 964–966.—The nature of the diffi- tly volatile compounds formed in the acetylene - flame is studied. With soln. containing NO_3^- se are oxides, *e.g.*, Y_2O. With soln. containing $_4^{2-}$, Er and Y give $M(SO_4)_3$, and Eu and Yb e MSO_4, where M is the metal. With soln. ntaining PO_4^{3-}, Eu, Yb and Y give MPO_4.

G. S. Smith

1432. **Determination of lanthanum, cerium, seodymium and neodymium as major com- nents by X-ray emission spectroscopy.** D. R. neval and H. L. Lovell (Dept. of Mineral paration, Pennsylvania State Univ., Pa., U.S.A.). *al. Chem.*, 1960, **32** (10), 1289–1292.—Discs for determination of La, Ce, Pr and Nd are pre- red by fusion of the sample with sodium borate; ndard discs are prepared similarly from the re rare-earth oxides. This procedure is accurate, edy and convenient, and little interference from ner rare-earth oxides is encountered. The alyses of four samples of widely varying compo- ion showed satisfactory accuracy.

K. A. Proctor

1433. **Successive complexometric determination thorium and total rare-earth metals.** Yu. A. ernikhov, R. S. Tramm and K. S. Pevzner. *vod. Lab.*, 1960, **26** (8), 921–924.—The soln. 5 ml), containing Th, rare-earth metals, Al, Mn, e, Ca, Sr, Nb and Ti, is mixed with 100 ml of ater, 1 ml of 5% sulphosalicylic acid soln. and 5 ml of 2% ascorbic acid soln. and treated with ..NH_3 until the pH is 1·5. After addition of 10 ml HCl - KCl buffer soln. (pH 1·6) and 0·5 ml of lenol orange or Alizarin red S - methylene blue ln. the cold soln. is titrated with 0·02M-EDTA to ve the content of Th. The titrated soln. is then eated with aq. NH_3 until the pH is 4, 10 ml of buffer soln. (pH 4·5) is added together with 5 ml of indicator soln. as before, and the boiling ln. is titrated with 0·02M-EDTA (disodium salt) give the content of rare-earth metals plus Mn. he content of Mn is determined separately and a rrection (0·09 ml of 0·02M-EDTA for each mg of n) is made.

G. S. Smith

1434. **Ion-exchange separation and spectrographic termination of some rare earths in beryllium, anium, zirconium and titanium metals, alloys d oxides. Collection with calcium and magnesium oride and ion-exchange separation.** S. Kallmann, . K. Oberthin and J. O. Hibbits (Ledoux and Co., eaneck, N.J., U.S.A.). *Anal. Chem.*, 1960, **32** 0), 1278–1281.—*Procedure*—The sample (10 to 50 g) is dissolved in aq. HF and the acid concn. is raised to $\simeq$ 2·5M by the addition of more HF. Yttrium chloride carrier is added ($\equiv$ 15 mg of Y_2O_3) and a soln. of Ca and Mg (0·25 g of CaO and 0·25 g of MgO dissolved in 10 ml of 6N-HCl, evaporated to dryness and dissolved in 20 ml of H_2O). The mixed fluorides are pptd. by heating for 1 hr. on a steam bath and setting aside for 3 hr. The ppt. is filtered off, ignited, converted into sulphate, and dissolved in N-HCl; any residual Be, U, Ca and Mg are separated from the rare-earth metals on a cation-exchange or mixed cation - anion exchange column. The Y and rare-earth metals are eluted with 12N-HCl, pptd. as oxalates, ignited to oxides and weighed, and the ratio of rare-earth metals to Y is determined spectrographically. From the known added weight of Y_2O_3 and the total yield of mixed oxides the original rare-earth-metal content of the sample can be calculated.

T. R. Andrew

1435. **Investigation of the spectrographic analysis of high-purity rare-earth oxides.** Yoshio Garrick Ishida (Kentucky Univ., Lexington, U.S.A.). *U.S. Atomic Energy Comm.*, Rep. ORO-279, 1959. 79 pp.—An investigation of the spectrographic analysis of high-purity rare earths containing trace amounts of other rare earths was made. The study was conducted in a controlled atmosphere of O - Ar (1:4), with d.c. arc excitation. Working curves and limits of detection were determined for Dy, Gd and Tb in an yttrium matrix; for Er, Lu and Tm in an ytterbium matrix; for Dy, Er and Y in a holmium matrix; and for Pr, Sm and Eu in a neodymium matrix. The minimum detectabilities of Y, Tb and Ho were found in a dysprosium matrix. The concn. of the impurities covered in the working curves ranged from 0·005 to 2·00%. The average precision of the determinations was $< 8\%$. The order of volatility of the rare-earth oxides in the O- Ar atmosphere was observed. The volatility was found to increase as the heats of vaporisation of the rare-earth metals decreased. Neodymium was the only rare-earth metal studied that showed a discrepancy in the correlation between volatility and heat of vaporisation. Working curves and limits of detection were determined for Dy and Ho employing a copper electrode - interrupted discharge method. Concn. from 1 μg to 60 μg per ml of the rare-earth oxides were used in the construction of the working curves. The average precision was $< 5\%$ for the working curves. A proposed method of calculating concn. ratios was tested and gave good accuracy in determining the relative amounts of Dy and Ho present in a series of samples. The sensitivities of the controlled atmosphere - d.c. arc and copper electrode - interrupted discharge method were compared. The sensitivity in detecting absolute amounts of rare-earth metals was of the same magnitude for the two methods. In terms of relative amounts, the sensitivity of detection of the d.c. arc method was superior to that of the copper electrode method.

Nucl. Sci. Abstr.

1436. **Photometric determination of cerium by means of o-tolidine.** N. Iordanov and K. Daiev (Sofia Univ., Bulgaria). *Zhur. Anal. Khim.*, 1960, **15** (4), 443–445.—*o*-Tolidine (**I**) is a selective and sensitive reagent for the determination of Ce. Add $La(NO_3)_3$ soln. as collector (1 ml) (12 mg per 10 to 200 μg of Ce) to the sample soln., dilute to

7 to 8 ml, precipitate La plus Ce with an equal vol. of satd. cold oxalic acid soln., and set the mixture aside till the next day. Then filter off, dry, and ignite the ppt. at 800° to 900°. Dissolve the residue in hot conc. H_2SO_4 (10 to 12 drops) and to the anhyd. yellow $Ce(SO_4)_2$ obtained add 0·05% **I** soln. in 10% H_2SO_4 (1 ml). Dilute the soln. to 100 ml and measure the extinction at 410 mμ. Beer's law is obeyed for conc. up to 3 μg of Ce per ml. The mol. extinction coeff. is ≃ 9500. The reaction with **I** is 20 times as sensitive as that with H_2O_2, and the determination is possible in the presence of Th, rare-earth metals and large amounts of Fe, Al, Mn and Ti. The method can be modified for the determination of Ce in apatite, titanite and monzonite. K. R. Cook

1437. **Spectrographic determination of yttrium in coal ash.** N. V. Arnautov and L. D. Shipilov. *Geologiya i Geofizika*, 1960, (1), 115–117; *Ref. Zhur., Khim.*, 1960, (18), Abstr. No. 73,066.—*Procedure*—Mix the sample with powdered carbon (1:1) and place in a cavity (diam. 3 mm, depth 3·5 mm, wall thickness 0·7 mm) in a carbon electrode. Excite the spectra in an a.c. arc at 9 amp. with a carbon upper electrode, and photograph in a medium Hilger spectrograph with a slit-width of 0·015 mm. Construct a calibration curve of log (I_Y/I_b) *vs.* log *C* (b = background) from the line 3327·87 A in the concn. range 0·001 to 0·4%. The reproducibility is within 10%. Prepare standards by adding Y_2O_3 to the ash. The concn. of Y in ash ranges from 0·002 to 0·02%. C. D. Kopkin

1438. **Rapid analysis of erbium by neutron activation followed by gamma-ray spectrometry.** Minoru Okada (Gov. Chem. Ind. Res. Inst., Tokyo). *Nature*, 1960, **188**, 52.—Place the sample (1 to 1000 mg), sealed in a polyethylene capsule, for 3 sec. in a neutron flux of 4×10^{10} per sq. cm per sec., cool for 5 sec., count for 3 sec., cool again for 2 sec. and count for 3 sec. Measure the pulse height of the peak due to the 0·208-MeV γ-radiation of ^{167m}Er. The coeff. of variation, estimated by the use of 7 standard samples, is ≃ 10%. Most of the variation seems to originate from error in time measurement. A. R. Rogers

1439. **Coulometric determination of europium and ytterbium at controlled potential.** E. N. Wise and E. J. Cokal (Chem. Dept., Univ., Tucson, Arizona, U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1417–1419.—Both Eu^{III} and Yb^{III} are reduced with nearly 100% current efficiency in 0·1M-tetraethylammonium bromide in methanol. The $E_{\frac{1}{2}}$ = 0·0 and − 0·78 V *vs.* the methanolic silver - silver bromide electrode. In samples containing both Eu and Yb, the sum only can be determined at − 1·2 V. The Eu is then determined by the method of Shults (*Ibid.*, 1959, **31**, 1095) and Yb is found by difference. The accuracy for 0·5 to 20 micro-equiv. is within 0·05 micro-equiv., but Nd and, to a less extent Sm, interfere with the determination of Yb. J. P. Stern

1440. **Determination of carbon on non-conducting catalysts by induction heating - conductimetric method.** R. J. Fox, J. W. Robinson and E. W. Seefield (Esso Res. Lab., Div. of Humble Oil and Refining Co., Baton Rouge, La., U.S.A.). *Anal. Chim. Acta*, 1960, **23** (4), 328–332 (in English).—The sample, containing ≃ 0·2 mg of C, is mixed with tin powder and iron chips and heated in a commercial h.f. induction furnace (Lindberg LI–501F). The CO_2 evolved is absorbed Ba(OH)$_2$ soln. in a commercial conductimet carbon-determination apparatus (Leco 515). T standard deviation is stated to be ± 0·007 at t 0·07% level of C (15 determinations). T. R. Andrew

1441. **Displacement technique for the co taminant-free precipitation of carbon dioxide barium carbonate.** J. Goodman and J. Steigm (Polytech. Inst., Brooklyn, N.Y., U.S.A.). *Chem Analyst*, 1960, **49** (3), 86.—The apparatus permi the pptn. of CO_2 as $BaCO_3$ without clogging of t bubbler, or exposure to the atmosphere. It readily constructed from a 150-ml conical flas two ground glass joints and a medium-porosi filter-crucible. P. D. Parr-Richard

1442. **Determination of carbon disulphide in t reaction mixture for the synthesis of xanthat** E. M. Idel'son. *Zavod. Lab.*, 1960, **26** (8), 947–948.- The CS_2 is extracted from the reaction mixtu with benzene and the extract is treated wi alcoholic KOH soln. The xanthate that is form is extracted with water, the soln. is treated wi acetic acid, and the neutral soln. is titrated wi 0·05N-iodine in the presence of starch. Altern tively, the benzene extract is treated with diethy amine and the colour is compared with that standards. G. S. Smith

1443. **Detection of carbonyl sulphide.** H Gamsjäger (Montanist. Hochsch., Leoben, Austria *Mikrochim. Acta*, 1960, (5–6), 758–761 (in Germa —The catalysis of the iodine - azide reaction used for the detection of COS. The limit of dete tion is between 25 and 50 μg. The COS may b concentrated either by absorption in ethanol KOH and regeneration by acidification, when t sensitivity is increased to 5 μg, or in acid PdC soln., when PdS is formed; COS may be detecte in 99·6% pure CO_2. Other sulphides, especiall H_2S and CS_2, interfere. J. P. Stern

1444. **Photometric determination of silicon γ-molybdosilicic acid.** W. Kemula and S. Rosołows (Dept. Inorg. Chem., Univ., Warsaw, Poland *Chem. Anal., Warsaw*, 1960, **5** (3), 419–428.—C boiling the acid soln. (pH 0 to 4) of α- an β-molybdosilicic acids for 30 min., a γ-molybd silicic acid is formed, to which the formul $H_4[Si(Mo_3O_{10})_4].nH_2O$ is ascribed. As this for is more stable (instability constant = $2·0 \times 10^{-1}$ it is more suitable for the colorimetric determin tion of silicon as the yellow complex at 420 m since critical time and pH effects are eliminate it is also suitable for the determination of Si in t presence of phosphates, since it is not affected b the large excess of citric acid used to decompose t molybdophosphate complex. P. Brych

1445. **Trace analysis of high-purity silicon.** F. Pohl and W. Bonsels (A.E.G. Res. Inst., Frankfur Main). *Mikrochim. Acta*, 1960, (5–6), 641–649 German).—Traces down to 1 p.p.m. of Pb, Cd, F In, Cu, Ni, Tl, Bi and Zn in semi-conductor silic are determined polarographically in a Souther Instruments K 1000 polarograph with errors 20%. Factors influencing accuracy and sensitivit include the method of pulverisation, the avoidan of inorganic reagents and working in a close system. A micro-cell of 0·2-ml capacity is illu trated. *Procedure*—Dissolve the sample (1 g) 100° in 40% HF (15 ml, added dropwise), in presen

$HClO_4$ (0·5 ml) and HNO_3 (5 ml), and remove e acids and SiF_4 at 150°. Oxidise the dry residue th 30% H_2O_2 and extract the Fe and Tl into ppropyl ether (3 × 1 ml). Remove organic matter om both phases by evaporation and treatment th H_2SO_4 (1 drop) and $HClO_4$ (0·2 ml) at 200°. ssolve the residue of the acid phase in 6N-aq. H_3 (0·2 ml) and polarograph in an atmosphere H, determining Cu at − 0·42 V, Cd at − 0·70 V, i at − 0·98 V and Zn at − 1·27 V; dissolve the id residue from a parallel run in 0·5M-Na tartrate ·2 ml) at pH 4·5 and determine Bi at − 0·49 V, ɔ at − 0·71 V, In at − 0·82 V and Zn at − 1·49 . Warm the residue from the organic phase with N-H_2SO_4 (3 drops) and 1% aq. hydrazine sulphate drop), evaporate to dryness, and dissolve the sidue in 0·5M-ammonium tartrate in N-aq. NH_3 ·2 ml); determine Tl at − 0·36 V and Fe at - 1·39 V. J. P. Stern

1446. **Quantitative spectrochemical analysis of inor elements in silicates with double internal andards.** H. Hamaguchi, R. Kuroda and R. egishi (Chem. Dept., Univ. of Education, Koishi-awa, Tokyo). *Bull. Chem. Soc. Japan*, 1960, **33** 7), 901–905.—The use of Sb_2O_3 (**I**) and palladium lack (**II**) as internal standards for volatile and on-volatile elements, respectively, in the spectro-raphic analysis of silicates, is described. **I** was sed for determining Ga, Pb and Sn, and **II** for Ni, o, Cr, V and Sc. **I** and **II** were mixed with carbon owder as buffer to give concn. of 0·8% (for **I**) and ·04% (for **II**). Selected internal standards and nalysis lines, with their excitation potentials and nalysis ranges, are listed. E. M. Mayes

1447. **Spectrographic determination of germanium nd gallium in coal.** A. N. Bronshteĭn, T. I. endul'skaya and M. Ya. Shpirt (Inst. of Mineral uels, Acad. Sci., USSR). *Zavod. Lab.*, 1960, **26** 8), 973–974.—The finely divided material is ashed with free access of air at 600° and mixed with an qual wt. of a buffer mixture containing carbon owder and NaCl (1 : 1) with 0·1% of SnO_2 to provide n internal standard. The spectrum of the mixture 20 mg) in a carbon electrode is excited by means of an a.c. arc (8 amp.), and the lines Ge 2651·18, Ga 2943·64, Sn 3034·12 and Sn 2839·99 A are neasured. The method is suitable for contents etween 0·001 and 1%. In the presence of > 0·1% of Ge or Ga the sample is diluted with SiO_2.
G. S. Smith

1448. **Determination of small amounts of aluminium in tin.** M. I. Shvaĭger and É. I. Rudenko (Magnitogorsk Metallurgical Combine). *Zavod. Lab.*, 1960, **26** (8), 939.—The sample (1 g) is dissolved in aqua regia and the soln. is evaporated several times with additions of HCl to remove Sn as $SnCl_4$. The residue is treated with 1 to 2 ml of H_2SO_4 (1 : 1), the soln. is evaporated, 2 ml of HCl (1 : 10) is added, and the soln. is again evaporated. The residue is dissolved in 2 ml of HCl (1 : 10), 0·85 ml of 10% NaOH soln. is added, and the soln. is diluted to 25 ml. An aliquot (2·5 ml) is treated with 0·2 ml of 5% ascorbic acid soln., 0·3 ml of 0·3% Chrome azurol S (C.I. Mordant Blue 29) (*cf.* Kashkovskaya and Mustafin, *Ibid.*, 1958, **24**, 1189) and 7 ml of an acetate buffer soln. (pH 5·37). After 5 min. the extinction is measured with a green filter. G. S. Smith

1449. **The use of dithizone as internal indicator in precipitation titrimetry. II. Direct volumetric determination of lead as lead phosphate.** M. Vancea and M. Volușniuc (Inst. Chem., Acad. R.P.R., Cluj, Romania). *Stud. Cercet. Chim., Cluj.*, 1959, **10** (2), 275–278.—Lead acetate soln. (≃ 0·1N) (10 to 20 ml) is added to acetic acid - Na acetate buffer (pH 4·5) (5 to 10 ml) and 1 ml of dithizone soln. in $CHCl_3$. The soln. is titrated with 0·1N-KH_2PO_4 until the brick-red colour changes to green. N. E.

1450. **Determination of traces of lead in prehistoric alloys. The use of a corundum rod as a scraping and crushing device.** H. Ballczo (Anorg. u. Phys. Chem. Inst., Univ. Wien, Austria). *Mikrochim. Acta*, 1960, (5–6), 973–982 (in German).—A small corundum rod is used to scrape off a 2-mg sample with minimum damage to the test-piece. The sample is dissolved in HNO_3 and evaporated almost to dryness; a second evaporation precipitates metastannic acid. After neutralisation with NH_3 vapour, the salts are dissolved in Ni - EDTA soln. and transferred to paper impregnated with Na_2SO_4. After treatment of the paper with tartrate buffer (pH 2·8), $PbSO_4$ is detected with Na rhodizonate. Large amounts of Cu and Sn do not interfere. P. D. Parr-Richard

1451. **Apparatus for the automatic determination of lead in ores and concentrates.** A. A. Khaĭdarov and A. P. Novikov. U.S.S.R. Pat. 127,473 (25th March, 1960).—The apparatus is designed for measurement with photo-electric multipliers, by the compensation method, of the bremsstrahlung γ-radiation caused by the lead in the sample and a standard when both are simultaneously irradiated with β-rays. The apparatus has a cathode follower with an intermediate amplifier in each arm of the bridge circuit. C. D. Kopkin

1452. **Determination of lead and tin in lead - tin solder alloys by controlled-potential electrolysis.** B. Alfonsi (Fiat Lab., Turin, Italy). *Anal. Chim. Acta*, 1960, **23** (4), 375–380 (in English).—Dissolve the sample (1 g) in 2 g of $K_2S_2O_7$ and 15 ml of H_2SO_4, cool, add HCl (1 : 3) (25 ml), $KMnO_4$ soln. (5%) (10 to 12 drops), tartaric acid (8 g in 25 ml) and aq. NH_3 until the soln. is clear (pH 6 to 8·5). Add succinic acid (2 g) and, slowly, hydrazine dihydrochloride (1 g). Adjust the pH to 7·2 to 7·3 or, if necessary to keep $PbSO_4$ in soln., to the value needed to produce a clear soln. (> 8·5). Deposit Pb (with some Cu, Bi and Ag) on a copper-plated platinum electrode at a potential of − 0·65 V *vs.* the S.C.E. until the current falls to ≃ 0·05 amp., when the pH should be adjusted to 5 and electrolysis continued till the current falls to 0·01 amp. After removal of the lead-plated electrode, add HCl (30 ml) and hydrazine dihydrochloride (0·2 g) to the soln. and electrolyse at > 1 amp., and finally at − 0·65 V *vs.* the S.C.E. The determination of both Pb and Sn may be completed in less than 3 hr. Results for Pb (0·1 to 0·8 g) are within ± 1 mg and for Sn (0·1 to 0·8 g) are within ± 0·5 mg of the amounts added. T. R. Andrew

1453. **Flame-spectrophotometric determination of calcium in lead metal.** R. E. Barringer (Union Carbide Nuclear Co., Y–12 Plant, Oak Ridge, Tenn., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. Y–1295, 1960. 19 pp.—A flame-spectrophotometric method for the determination of Ca in lead metal in the range of 0·02 to 0·1% was developed. A minimum of sample preparation and time is

required. Measurement of the Ca emission at 554 mμ gave limits of error ($p = 0{\cdot}95$) of ± 4·2%. NUCL. SCI. ABSTR.

1454. **Arsenate method for the iodimetric determination of titanium in the presence of other cations.** G. B. Shakhtakhtinskiĭ and A. M. Mukimov. *Azerbaidzh. Khim. Zhur.*, 1959, (3), 59–63; *Ref. Zhur., Khim.*, 1960, (15), Abstr. No. 61,054.—Precipitate Ti^{4+} from an acetic acid medium containing 1% of free H_2SO_4 with Na_2AsO_4; Ag^+, Hg^{2+}, Cu^{2+} and Cd^{2+} do not interfere. Dissolve the ppt. in H_2SO_4 and titrate the liberated H_3AsO_4 iodimetrically; Bi^{3+}, Sb^{3+}, Sn^{2+} and Sn^{4+} are co-pptd. with Ti and lead to high results. When titanium arsenate is pptd. from 3% HNO_3 soln. in the absence of H_2SO_4, much less Pb is co-pptd. than from 1% HNO_3 soln. K. R. COOK

1455. **Separation of titanium from manganese by the ion-exchange method.** I. K. Tsitovich (Kuban Agric. Inst., Krasnodar). *Zhur. Anal. Khim.*, 1960, **15** (4), 503–504.—The distribution coeff. of $[TiO(H_2O_2)]^{2+}$ and MnO_4^- between the cationites KU-1, KU-2 and SBS and HCl soln. (0·1 to 10N) were studied and results are tabulated. To separate Ti from Mn when they are in the ratios of 1:1000 to 1000:1 use a column (height 10 cm, diameter 1 cm) filled with cationite KU-1 (H^+ form). Wash it with 0·5N-HCl, pour in the test soln. (0·5N with respect to HCl), elute Mn with 0·5N-HCl at the rate of 30 drops per min., and elute Ti with 4N-HCl. Titanium forms anion complexes in > 8N HCl soln. K. R. COOK

1456. **Photometric determination of zirconium as the molybdophospho-zirconium complex.** R. M. Veĭtsman (All-Union Sci. Res. Inst. of Hard Alloys). *Zavod. Lab.*, 1960, **26** (8), 927–929.—The acid (H_2SO_4) soln. (20 ml) containing from 1 to 25 mg of Zr is mixed with 10 ml of 10% K citrate soln., then neutralised to Congo red paper with aq. NH_3, and diluted to 200 ml. An aliquot (20 ml) is treated with 20 ml of water, 0·5 ml of 5% $(NH_4)_2HPO_4$ soln. and 3 ml of 10% $(NH_4)_2MoO_4$ soln. and then neutralised with H_2SO_4 (1:4), with Congo red paper as indicator. The soln. is diluted to 100 ml and set aside for 10 to 15 min. An aliquot (20 ml) of the yellow soln. is treated with 15 ml of 4% EDTA (disodium salt) soln. The time is noted and 10 ml of H_2SO_4 (1:4) is added at once, followed after 30 sec. by 1 ml of 0·1% $SnCl_2$ soln. After being mixed for 2 to 5 sec. the soln. is diluted to 100 ml, and the extinction is measured with a red filter. The content is calculated by means of a calibration curve. G. S. SMITH

1457. **Radiochemical separation of zirconium-95 and niobium-95 with tri-n-butylphosphine oxide.** Hirokazu Umezawa and Reinosuke Hara (Japan Atomic Energy Res. Inst., Tokai, Ibaraki-ken). *Anal. Chim. Acta*, 1960, **23** (3), 267–270 (in English). —A separation scheme is presented for ^{95}Zr and its daughter ^{95}Nb, based on the extraction of the ^{95}Zr into 0·005M-tri-n-butylphosphine oxide in CCl_4 from 2N-HNO_3. The ^{95}Nb remains in the aq. phase. The recovery of ^{95}Zr is 95%. Separation from HCl soln. was not possible. H. M.

1458. **Rapid chromatographic semi-quantitative micro-determination of total zirconium and hafnium in sulphuric and oxalic acid media.** A. Górski and J. Moszczyńska (Dept. of Metallurgy, Inst. of Technol., Warsaw). *Chem. Anal., Warsaw*, 1960, **5** (3), 395–400.—The method is suitable as a production control test, and can be used for detecting 6 μg of Zr plus Hf in 1 ml of the strongly acid eluates from ion-exchange columns. The chromatograms are prepared on columns (100 mm × 4 mm) packed with acid-washed alumina by the dropwise addition of 0·1 ml of 0·15% Alizarin red S soln. followed by 0·1 ml of the sample. The intensity and position of the brown rings given by the metal - dye complexes depends on the concn. of the metals, and a semi-quant. estimation is obtained by comparison with freshly prepared standards or a permanent colour chart. P. BRYCH

1459. **Spectrophotometric determination of zirconium in magnesium-base alloy.** Shozo Shibata, Yoshio Ishiguro and Teiichi Matsumae (Gov. Ind. Res. Inst., Nagoya, Japan). *Anal. Chim. Acta*, 1960, **23** (4), 384–387 (in English).—Arsenazo reacts with Zr in acid soln. (pH 1·6 to 2·0) to give a blue colour (max. absorption at 578 mμ), which obeys Beer's law up to at least 20 p.p.m. of Zr (extinction ≃ 0·5 at 578 mμ in a 1-cm cell). Aluminium, Mg, Cu, Fe, Ce, Ti, U, acetate, SO_4^{2-}, Cl^- and NO_3^- do not interfere; Th, Nb, Ta, citrate, oxalate, tartrate, PO_4^{3-} and ClO_4^- must be absent. The technique has been applied to the determination of Zr (0·01 to 1%) in magnesium alloys. T. R. ANDREW

1460. **Colorimetric determination of hafnium oxide in admixture with zirconium oxide.** L. I. Kononenko, R. S. Lauer and N. S. Poluéktov. *Ukr. Khim. Zhur.*, 1959, **25** (5), 633–638; *Ref. Zhur., Khim.*, 1960, (12), Abstr. No. 47,061.—The coloured complexes of Zr and Hf with arsenazo (**I**) behave differently as the HCl concn. of the soln. is increased, the complex of Hf being decolorised to a greater extent than that of Zr. In N-HCl, the extinction of a soln. of the Hf complex is one-nineteenth, and that of the Zr complex one-half, that in 0·25N-HCl, so that by measuring the extinctions of a soln. of the complex with **I** at 0·25N and at 1·0N with respect to HCl, the content of Hf in a mixture with Zr can be determined. The different behaviour with respect to increasing HCl concn. may be due to the different degrees of dissociation of the Zr and Hf complexes, the apparent dissociation constants of which are 0·12 × 10^{-10} and 0·86 × 10^{-10}, respectively. *Procedure*—Dissolve 15 mg of the sample of mixed HfO_2 and ZrO_2 in 2 to 3 ml of HF, evaporate the soln. with 2 ml of conc. $HClO_4$ and 10 ml of a 4% H_3BO_3 soln. (to remove F^-), dissolve the residue in N-HCl and make up to 250 ml with N-HCl. Take two 5-ml aliquots of the soln., add to each 30 ml of water, to the first add 1·88 ml and to the second add 11·25 ml of 4N-HCl; add to each soln. 2·5 ml of 1% gelatin soln. and 5 ml of 0·1% **I** soln., make up to 50 ml and measure the extinctions at 570 mμ in 1-cm cells against a reagent blank. Calculate the ratio of the extinctions in 0·25N- and 1·0N-HCl and obtain the HfO_2 content from a calibration curve. The method can be used to determine from 20% upwards of HfO_2 in mixtures of HfO_2 and ZrO_2 with an abs. error ranging from − 4·0 to + 6·0%. C. D. KOPKIN

1461. **Arsenate method for the iodimetric determination of thorium.** G. B. Shakhtakhtinskiĭ and I. A Mamedov. *Azerbaidzh. Khim. Zhur.*, 1959, (2), 105–110: *Ref. Zhur., Khim.*, 1960, (15), Abstr. No. 61,059.—*Proceedure*—Heat the test soln. (15 ml

boiling, and add a slight excess of aq. NH_3 soln. :5). Add boiling 0·5N-Na_3AsO_4 (15 ml) neutra- ed to methyl red and boil the soln. and ppt. $ThOHAsO_4$) for $\simeq$ 10 min. Filter through a ntered-glass crucible (No. 4), wash the ppt. (× 7) ith hot H_2O and dissolve it in H_2SO_4 (2:5) (25 to ml). Add benzene (20 to 25 ml) and 2N-KI ml), stir for a few seconds, dilute the aq. layer ith an equal vol. of H_2O and titrate with $a_2S_2O_3$ soln. until the benzene layer is colourless; ml of 0·1N-$Na_2S_2O_3$ $\equiv$ 0·0116 g of Th.

K. R. Cook

1462. **Estimation and separation of thorium with hydroxytartaric acid.** G. R. Amladi and Y. V. awande (St. Xavier's Coll., Bombay). *J. Univ. ombay*, 1960, **27**, 24–28.—Thorium nitrate is uant. pptd. from acid soln. with 2% dihydroxy- rtaric acid soln. (**I**) above pH 0·6; La and Ce re also pptd. by **I**, but from less acid soln. horium in mixtures with lanthanum and cerium itrates (1:4:4) is separated by adjusting the pH 0·7 to 1·0, treating with **I** to precipitate Th, -dissolving in conc. HNO_3, evaporating, extrac- ng with water, re-adjusting the pH to 0·7 to 1·0, nd re-pptg. with **I**. **I** gives as good results in the xtraction of Th from monazite sands as one of the sual reagents, *e.g.*, *m*-nitrobenzoic acid.

Chem. Abstr.

1463. **Thorium determinations with 8-hydroxy- uinaldine.** A. Corsini and R. P. Graham (Burke hem. Lab., McMaster Univ., Hamilton, Canada). *nal. Chim. Acta*, 1960, **23** (3), 248–256.—The etermination of Th with 8-hydroxyquinaldine (**I**) as been studied and the essential conditions have een established. (*i*) A 70% excess of **I** must be resent if the ppt. is to be only the 1:4 chelate. or concn. of Th $<$ 0·7 mg per ml the excess hould be as for 0·7 mg per ml; this can be achieved y using a fixed amount of **I** and keeping the Th oncn. below 0·7 mg per ml. (*ii*) The pH must e between 6·1 and 11·5, and the manner of adding he aq. NH_3 used to give this pH is important. he concn. of aq. NH_3 must not exceed 1·0M and it ust be added dropwise with very vigorous stirring. this is not done the ppt. will consist partly of ydrated ThO_2. (*iii*) The ppt. should be digested t 70° for $\not<$ 1 hr., in order to convert any small mounts of hydrated ThO_2 into the chelate. The mal determination of Th can be carried out romimetrically, or gravimetrically by drying he ppt. for 1 hr. at 110° and weighing it as $h(C_{10}H_8ON)_4$.

H. M.

1464. **Derivatives of 1,4-dihydroxyanthraquinone s metal indicators in the titration of thorium with isodium EDTA.** E. G. Owens, II, and J. H. Yoe Pratt Trace Analysis Lab., Univ. of Virginia, harlottesville, U.S.A.). *Anal. Chim. Acta*, 1960, **3** (4), 321–324 (in English).—2-Phenoxyquini- arin-3,4′-disulphonic acid (dipotassium salt) (**I**) and uinizarin-2-sulphonic acid (sodium salt) (**II**) are dvocated as having advantages over Alizarin red (**III**) in the complexometric titration of Th. uitable pH ranges for these indicators are— ·4 to 3·4 for **I**; 2·0 to 3·4 for **II**; and 2·2 to 3·4 for **III**. The colour changes at the end-point are harpest with **I**.

T. R. Andrew

1465. **A re-investigation of the determination of horium with SPADNS [sodium 4,5-dihydroxy-3- (4-sulphophenylazo)naphthalene-2,7-disulphonate].** . A. Cooper and M. J. Vernon (Dept. of Geo- hysics, Australian Nat. Univ., Canberra). *Anal. Chim. Acta*, 1960, **23** (4), 351–354 (in English).—Attempts to repeat the work of Banerjee (*Anal. Abstr.*, 1957, **4**, 2156) have proved unsuccessful, an extinction of 0·4 for 100 μg of Th per 25 ml being found as compared with the previously reported value of 0·4 for 4 μg of Th. The sensitivity is given as 0·011 μg of Th per sq. cm for log I_0/I = 0·001 at 580 mμ, while Banerjee quoted 0·012 μg of Th per sq. cm, indicating a discrepancy in the earlier work. The effects of reagent concn. and acetate buffer concn. are reported.

T. R. Andrew

1466. **Rapid determination of thorium by fluoride titration using some chromotropic [acid] azo dyes.** S. K. Datta [Victoria (Govt.) College, Coochbehar, India]. *Bull. Chem. Soc. Japan*, 1960, **33** (7), 882–884.—The titrimetric determination at pH 2·4 to 2·8 of up to 50 mg of Th per 50 ml of soln. with NaF, with 0·5 ml of 0·1% nitroso-SNADNS-4, SNADNS-6 and CHPADNS, is described. These dyes, which are prepared from 4-aminonaphthalene-1-sulphonic acid, 5-amino-naphthalene-2-sulphonic acid and 4-aminosalicylic acid, respectively, form pink to violet complexes with Th, which are decomposed by fluoride with a marked colour change. Interfering substances include iron, cerium, rare-earth metals and alkaline-earth metals, phosphates, borates, oxalates, tartrates and citrates.

E. M. Mayes

1467. **Photometric determination of thorium in minerals with arsenazo II.** S. B. Savvin, M. P. Volynets, Yu. A. Balashov and V. V. Bagreev (Vernadskiĭ Inst. of Geochem. and Anal. Chem., Acad. Sci., Moscow). *Zhur. Anal. Khim.*, 1960, **15** (4), 446–451.—The method is suitable for the determination of 10^{-2} to 10^{-4} % of Th in minerals. Two methods are recommended for the isolation of Th, namely, double fluoride pptn. and fluoride - oxalate pptn.

K. R. Cook

1468. **Analytical chemistry of thorium. II. Complexometric determination of thorium in monazite concentrates after its isolation on KU-2 cationite.** Yu. A. Chernikhov, V. F. Luk'yanov and A. B. Kozlova. *Zhur. Anal. Khim.*, 1960, **15** (4), 452–454.—*Procedure*—Fuse the monazite concentrate (2 g) with Na_2O_2 (10 to 12 g) at 600° to 650°, leach the melt with hot H_2O, filter off the residue and wash it with 5% NaOH soln., followed by hot HCl (1:1). Evaporate the soln. almost to dryness, add conc. HCl (20 ml) and H_2O (20 ml) and filter off the residue containing $ZrO_2(P_2O_5)_4$. Add 20% tartaric or trihydroxyglutaric acid soln. (5 ml) to the combined filtrate and washings and dilute to 100 ml. Pass a 25-ml aliquot of the soln. through a column (height 24 cm, diam. 1·5 cm) containing iron-free KU–2 resin (grain size 0·16 to 0·3 mm) (20 to 25 ml). Wash the column with 10 to 15-ml portions of 20% HCl (70 ml); the final percolate should be colourless. Then pass 20% NH_4Cl soln. (50 ml) through the column, and finally elute Th with 5 to 7-ml portions of 20% ammonium carbonate soln. (50 ml). Wash the column with H_2O (20 to 30 ml) and add the washings to the eluate. Add HCl (1:1) to the combined soln. to decompose the ammonium carbonate, then boil the soln. for 2 to 3 min. Adjust the pH of the soln. with aq. NH_3 soln. to 2·4 to 2·6 (to 2,4-dinitrophenol), add 0·5% aq. xylenol orange soln., and titrate with $\simeq$ 0·2M-EDTA to a lemon-yellow end-point. Good recoveries (tabulated) are obtained by this method.

K. R. Cook

1469. **Radiometric determination of thorium in tungsten and molybdenum.** L. N. Aleksandrov. *Zavod. Lab.*, 1960, **26** (8), 975–977.—A method based on measurement of the α-radiation intensity of ^{232}Th is described. G. S. SMITH

1470. **Differential electrolytic potentiometry. IV. Application to micro-coulometric titrimetry. The determination of hydrazine at microgram levels.** E. Bishop (Univ., Exeter, England). *Mikrochim. Acta*, 1960, (5–6), 803–815.—Hydrazine is titrated with electrically generated bromine in a micro-titration apparatus (illustrated). The accuracy for 1 to 3 μg of hydrazine with a generating current of 100 μA is within ± 0·3%, and on 0·2 and 0·02 μg with a current of 10 μA it is within ± 1% and ± 5%, respectively. J. P. STERN

1471. **Colorimetric method for the estimation of azide ions.** P. J. Staples (University Coll., London, England). *Chem. & Ind.*, 1960, (39), 1210–1211.—The amount present in 5 ml of a 10^{-3} to 10^{-4}M-soln. is determined by using the reaction between N_3^- and NO_2^-. This reaction is rapid in acid and especially in acetic acid - acetate buffer soln. Sulphanilic acid is then diazotised with the excess of NO_2^-, and the diazonium salt is coupled with **1**-naphthylamine to form a water-soluble stable orange - yellow dye, the extinction of which is measured at 475 mμ. Results are referred to a calibration curve. The error is 1%. When co-ordinated azido-groups are present they interfere by reacting with NO_2^-. The determination can then be made on the percolate after passage of the sample soln. through an ion-exchange column of IR-120 (Na^+ form). It is also possible to titrate N_3^- (≮ 10^{-2}M) potentiometrically with standard $AgNO_3$ soln. R. M. S. HALL

1472. **Analytical uses of bromine chloride. Determination of hydroxylamine.** K. Burger, F. Gaizer and E. Schulek (L. Eötvös Univ., Budapest, Hungary). *Talanta*, 1960, **5** (2), 97–101 (in English).—Hydroxylamine is determined by oxidation with BrCl produced by the action of HCl on a soln. containing equivalent amounts of $KBrO_3$ and KBr. *Procedure*—To the sample soln. (containing **3** to **12** mg of hydroxyammonium chloride) add sufficient 0·1N-$KBrO_3$ (2·7835 g of $KBrO_3$ and 3·9670 g of KBr per litre) to give ≮ 100% excess of BrCl on acidification. Dilute to 40 ml with water, add 20% HCl (5 to 10 ml) and set aside for 5 min. Add 5% KI soln. (10 ml) and titrate with 0·1N-$Na_2S_2O_3$. The error of the method is ± 0·5%. It is shown that in the method of Kurtenacker and Wagner (*Z. anorg. Chem.*, 1922, **120**, 261) the effective oxidant is a mixture of BrCl and Cl. W. T. CARTER

1473. **Determination of ammonia in the presence of volatile amines.** N. Bellen and Z. Bellen (Anal. Dept., Inst. of Gen. Chem., Warsaw, Poland). *Chem. Anal., Warsaw*, 1960, **5** (3), 461–469.—The sample soln. (containing NH_3 equiv. to 0·1 to 0·35 g of NH_4Cl) is digested for 30 min. with sodium cobaltinitrite (25 g) and $NaNO_3$ (25 g) in acetate buffer (pH 4·8) (100 ml). The pptd. sodium ammonium cobaltinitrite is filtered off, washed with acetate buffer satd. with sodium ammonium cobaltniitrite (5 × 5 ml), and decomposed with NaOH. The liberated NH_3 is distilled with 0·2N-HCl, the excess of which is titrated with 0·2N-NaOH. The accuracy is ± 1%. This method can be used in the presence of a tenfold excess of amines. P. BRYCH

1474. **Titrimetric determination of combine carbon dioxide in anhydrous ammonia.** A. K Adam, R. Syputa and W. E. Stephenson (Wester Operations Inc., Standard Oil Co. of California Richmond, U.S.A.). *Anal. Chem.*, 1960, **32** (10) 1319–1320.—The sample (≃ 500 ml) contained i a stainless-steel cylinder is allowed to evaporat spontaneously at a temp. maintained at < 70° F Combined CO_2 remains in the cylinder as ammoniur carbamate. The ammonium carbamate is de composed in the cylinder with 2N-H_2SO_4 and th evolved CO_2 is carried by a stream of N through scrubber containing 2N-H_2SO_4 and absorbed in a excess of standard $Ba(OH)_2$ soln., which is the back-titrated with oxalic acid soln. Results o synthetic mixtures containing 5 to 50 p.p.m. o CO_2 show deviations from the amounts added o ≯ 3 p.p.m. T. R. ANDREW

1475. **New method for the determination o nitrates.** P. W. West and G. L. Lyles (Coate Chem. Lab., Louisiana State Univ., Baton Rouge) *Anal. Chim. Acta*, 1960, **23** (3), 227–232.—Chromo tropic acid (**I**) in conc. H_2SO_4 reacts with nitrate to give a yellow colour, the intensity of which is measure of the nitrate concn. *Procedure*—The tes soln. (≯ 5 p.p.m. of NO_3^-) (3 ml) is added to 7 m of reagent soln. (0·01 g of **I** in 100 ml of conc H_2SO_4 containing 1 ml of conc. HCl) in a 10-m flask. When the soln. has cooled to room temp. i is diluted to volume, and the extinction is read a 357 mμ and referred to a calibration graph. Oxides of N in the atmosphere may be determined by drawing the air through 7 ml of the reagent and treating as described above. Bromate, Br^-, ClO_3^- Ti^{3+} and Ti^{4+} interfere seriously, Be^{2+}, Bi^{3+}, I^- IO_3^-, Fe^{2+}, Fe^{3+}, MoO_4^{2-} and Ru^{3+} only slightly Two calibration graphs must be prepared for the ranges 0·0 to 0·8 p.p.m. and 1·0 to 5·0 p.p.m. o NO_3^-, respectively. H. M.

1476. **Oxidimetric determination of small amounts of nitrate in sodium nitrite.** W. Leithe (Österr. Stickstoffwerke A.-G., Linz, Austria). *Mikrochim. Acta*, 1960, (5–6), 762–765 (in German).—After destruction of the NO_2^- with excess of hydrazine sulphate, the NO_3^- in sodium nitrite are determined by reaction with excess of 0·02N-$FeSO_4$ and back-titration with 0·01N-$K_2Cr_2O_7$. The method is used for NO_3^- contents as low as 0·05% and, for 0·2 to 1% of NO_3^-, involves errors ≯ 0·03% absolute. *Procedure*—Add the sample (1 g) in 50 ml of water to ice-cold 15% hydrazine sulphate soln. (25 ml) during 10 min. Set aside for 5 min. at room temp. and dilute to 100 ml. Add 10 ml of this NO_2^--free soln. to NaCl (2 g) and then add 0·02N-$FeSO_4$ (10 ml) and boil the soln. vigorously for 3 min. Cool the soln. and titrate the excess of $FeSO_4$ with 0·01N-$K_2Cr_2O_7$ in the presence of 0·0025M-ferroin (5 drops). Compare with a blank titrated at the same temp. J. P. STERN

1477. **Specific volumetric determination of nitrite.** H.-W. Kaloumenos (Friedrich Goetze A.-G., Burscheid bei Köln, Germany). *Werkst. u. Korrosion*, 1960, **11** (10), 626.—Sodium nitrite reacts quant. with sulphamic acid according to the equation—

$$NaNO_2 + H_2NSO_3H = NaHSO_4 + H_2O + N_2.$$

Procedure—To 20 ml of filtered test soln. add 150 ml of H_2O, 20 ml of 0·1N-sulphamic acid and 20 ml of conc. H_2SO_4. Warm the mixture to 60° ± 5° and back-titrate the excess of sulphamic acid potentiometrically with 0·1N-$NaNO_2$, at a rate

2 to 4 drops per sec., until the potential does not crease on further addition of $NaNO_2$ soln. The thod can be used for the analysis of pickling t and nitrous acid derivatives in dilute and ncentrated soln., and in the presence of organic vents. R. E. E.

1478. **Optical phenomena in turbidimetric analysis plied to sulphate determination in phosphoric acid.** E. Dahlgren (AB Förenade Superfosfatfabriker, ndskrona, Sweden). *Acta Chem. Scand.*, 1960, (6), 1279–1287.—Optimum measuring conions for turbidimetric analysis are discussed eoretically, but satisfactory experimental verifition of the theory could not be obtained with SO_4 dispersions in 0·5% gelatin soln. Conditions commended are that the wavelength should be 410 mμ (for determinations within the range 05 to 0·20 mg of S per 50 ml) and that concentran and cell size should be chosen so that an tinction of 0·3 to 0·5 is obtained. A coeff. of riation of 1·5% was obtained for 12 samples der these conditions. S. M. Marsh

1479. **Titrimetric determination of phosphorus apatite by means of ion-exchange resins.** A. ewandowski and H. Witkowski. *Prace Kom. at.-Przyr. Poznań. Tow. Przyj. Nauk*, 1959, **7**), 3–7; *Ref. Zhur., Khim.*, 1960, (12), Abstr. No. ,112.—The method used is based on the complexoetric titration of soluble phosphates in an mmoniacal medium, with Eriochrome black T as dicator [*cf. Ref. Zhur., Khim.*, 1956, (13), Abstr.). 39,860]. *Procedure*—Treat 1 g of the finely ound sample with a mixture of conc. HNO_3 and Cl (1:3) and evaporate to dryness. Add 0·5 ml conc. HCl and again evaporate to dryness, and peat to render silicic acid completely insoluble.) the residue add 1 or 2 ml of conc. HCl, dilute, ter, and make up to 100 ml. To remove interring elements, pass 20 ml of the soln. through a lumn containing 10 g of a phenolsulphonic resin [+ form) and wash with water to give a total olume of 100 to 120 ml; evaporate the soln. to or 20 ml, add 30 ml of buffer soln. (50 g of H_4Cl and 400 ml of conc. aq. NH_3 in 1 litre) and or 3 drops of Eriochrome black T soln., add andard $MgSO_4$ soln. (≃ 0·1M), set aside for 20 in., filter off the pptd. $MgNH_4PO_4$ and titrate e excess of $MgSO_4$ with 0·1M-EDTA (disodium lt). The results agree to within ≃ 1% with those the gravimetric method. C. D. Kopkin

1480. **Rapid determination of soluble phosnoric anhydride in open-hearth slags.** N. N. apin and N. S. Prilutskaya. *Sb. Nauch. Trud. idanovsk. Metallurg. Inst.*, 1960, (5), 387–392; *ef. Zhur., Khim.*, 1960, (18), Abstr. No. 73,114.—nosphorus compounds are dissolved in citric acid ith subsequent photometry as yellow molybdoanadophosphoric acid. *Procedure*—Add 5 ml of hanol to the sample of finely ground slag (2·5 g), ake up to 250 ml with 2% citric acid soln., ake mechanically for 15 min. and filter through dry filter, discarding the first 50 ml. Make up 0 ml of the filtrate to 500 ml; to 10 ml of the ln. add 40 ml of water and 30 ml of reagent ix equal vol. of HNO_3 (1:2), 25% NH_4VO_3 ln. and 5% ammonium molybdate soln.; set ide for 4 to 6 days before use]. Dilute to 100 ml d after 5 min. measure the extinction in a 20-mm ll with a blue filter. A single determination kes 25 min.; the relative error is ⩾ 1·6%. The esence of SiO_2 (0·7 to 0·8 mg) does not interfere. It is shown that all P-containing compounds found in open-hearth slag are soluble in 2% citric acid soln., which enables the value of the slag to be deduced from the result. C. D. Kopkin

1481. **Quantitative determination of the composition of Graham's salt.** A. Swinarski and M. Piotrowska (Dept. of Inorg. Chem., Copernicus Univ., Toruń, Poland). *Chem. Anal., Warsaw*, 1960, **5** (3), 435–443.—An investigation of the method of Jones (*Ind. Eng. Chem., Anal. Ed.*, 1942, **14**, 536) showed that pH values and digestion times significantly influence the results, and the following refinements are therefore proposed. The precipitation of polyphosphates with $BaCl_2$ should be carried out at pH 3·0, and the ppt. should be filtered off after digestion for 20 min. Trimetaphosphates are determined in the filtrate, and the separation of ortho- and pyro-phosphates with $BaCl_2$ should be carried out at pH 8, with digestion for 30 min. To avoid possible hydrolysis of polyphosphates, sample soln. should be prepared immediately before the analysis. These modifications were tested by analysing ten samples from each of two batches of Graham's salt, and the following mean errors (as % of sample) were obtained—total P_2O_5 0·08, 0·05; polyphosphate 0·43, 0·48; trimetaphosphate 0·51, 0·45; orthophosphate 0·65, 0·51; and pyrophosphate 0·30, 0·23. P. Brych

1482. **Detection of polyphosphates by paper chromatography.** F. Bukatsch. *Mikrokosmos*, 1959, **48**, 276–278.—The apparatus is described. Under favourable conditions satisfactory separation of the various phosphates is achieved in about 3 hr. After development the paper is sprayed with a soln. containing equal parts of 7·5% aq. ammonium molybdate soln. and conc. HNO_3. The zones of molybdophosphate thus produced are made visible by reduction to molybdenum blue in bright sunlight. With a 1-drop sample containing a concn. of 1% of each acid (sodium salt) the ultimate positions occupied on the chromatogram are as follows—near the top, where the sample was applied, hexaphosphoric acid; next in order are tetra- and tri-phosphoric acids, pyrophosphoric and orthophosphoric acid. Analysis of sausage and similar meat products can be carried out by using 1 drop of soln. obtained by extracting with glacial acetic acid or with 20% trichloroacetic acid. Chem. Abstr.

1483. **Chromatographic and electrophoretic analysis of commercial triphosphates and hexametaphosphates.** M. Lenzi and E. Mariana (Univ. Rome). *Rass. Chim.*, 1959, **11** (3), 11–15.—The chromatographic methods of Ebel and Volmar (*Compt. Rend.*, 1951, **233**, 415) and of Ebel (*Ibid.*, 1952, **234**, 621; Volmar *et al.*, *Ibid.*, 1952, **235**, 372) were found to be most suitable for the analysis of commercial triphosphates and allow a neat separation of di- and tri-phosphates. The technique of Grunze and Thilo (*Die Papierchromatographie der Kondensierten Phosphate*, Akademie-Verlag, Berlin, 1954) was found the most satisfactory; S. & S. 2043 paper gave the best results, and an acid medium (75 ml of isopropyl alcohol, 25 ml of H_2O, 5 g of trichloroacetic acid and 0·3 ml of 20% aq. NH_3) gave results superior to an alkaline medium (30 ml of isobutyl alcohol, 30 ml of 95% ethanol, 39 ml of H_2O and 1 ml of 25% aq. NH_3) at 20° for 25 hr. In the former case, the R_F values were—orthophosphate, 0·7; pyrophosphate, 0·42;

triphosphate, 0·31; trimetaphosphate, 0·26; tetraphosphate, 0·23; and tetrametaphosphate, 0·12; in the latter case, the R_F values were—trimetaphosphate, 0·71; tetrametaphosphate, 0·63; orthophosphate, 0·44; pyro- and tri-phosphate, 0·35; thus, the successive use of both methods in two-dimensional chromatography improves separation. Electrophoresis was carried out on Whatman No. 3 paper (9 cm × 45 cm), compressed between glass plates, with a potential difference of 1000 V for 1 hr. or more. At pH 8·5 (borax - H_3BO_3 - NaCl buffer) the displacement in cm per min. was 0·55 for trimeta-, 0·39 for tetrameta-, 0·43 for ortho-, 0·33 for tri- and tetra-, and 0·26 for pyro-phosphate. The lack of separation between the higher polyphosphates is not objectionable in the case of commercial triphosphates. CHEM. ABSTR.

1484. **Determination of small amounts of arsenic in organic matter.** The Metallic Impurities in Organic Matter Sub-Committee of The Analytical Methods Committee of The Society for Analytical Chemistry. *Analyst*, 1960, **85**, 629–643.—Two methods are recommended, each being preceded by destruction of the organic matter by wet oxidation. In the molybdenum blue method, the As is extracted with diethylammonium diethyldithiocarbamate soln. in $CHCl_3$ and converted into the molybdoarsenate complex, which is then reduced by means of hydrazine sulphate to a molybdenum blue complex for spectrophotometric determination at 840 mμ. In the second method, the As is separated by distillation as $AsCl_3$, and determined by a modification of the Gutzeit method. Collaborative work on the first method is described and the procedures for both methods are given in detail. The molybdenum blue method can give consistently accurate results for the As content of organic matter in the range 1·5 to 15 μg of As, and with specially purified reagents the lower limit of the range can be extended to 0·5 μg. Interference by Si and P is avoided and by Ge is unlikely. The Gutzeit method, being dependent on the assessment of a coloured stain, is recommended only as an approximation, and is particularly useful when it is necessary to determine whether or not the As content exceeds a certain limit.
A. O. JONES

1485. **Separation of radioactive arsenic from germanium irradiated with protons.** K. Saito, S. Ikeda and M. Saito (Inst. for Nuclear Study, Tokyo Univ., Tanashi). *Bull. Chem. Soc. Japan*, 1960, **33** (7), 884–887.—The separation of almost carrier-free arsenic from germanium irradiated with protons, by distillation and by solvent extraction with diethyldithiocarbamate, is described. Total arsenic recovery was 94% and > 90%, respectively. The use of an acetate buffer was not found essential. Impurities in the final product, including carrier arsenic, are less than 10 μg for 0·4 mC, the specific activity for ^{74}As being ≃ 1 mC per 25 μg immediately after preparation. The over-all yield for ^{74}As is ≃ 70%. E. M. MAYES

1486. **The use of dithizone as internal indicator in titrimetry by precipitation. V. Direct volumetric determination of arsenate as bismuth arsenate.** M. Vancea and M. Volușniuc (Inst. Chem., Acad. R.P.R. Cluj, Romania). *Stud. Cercet. Chim., Cluj*, 1959, **10** (2), 291–293.—The method is based on the titration of AsO_4^{3-} in the cold with a soln. of $Bi(NO_3)_3$ containing < 2% of free HNO_3, with dithizone as internal indicator, until the indicat changes from green to clear grey. H. SHER

1487. **New colorimetric applications of th chromate - o-dianisidine system. I. Indirect color metric determination of antimony(III), hyposulphit hydrazine, hydroxylamine and hydrogen peroxid** F. Buscaróns, J. Artigas and C. Rodriguez-Roc (Fac. of Sci., Univ., Barcelona, Spain). *An Chim. Acta*, 1960, **23** (3), 214–216 (in English).- The method consists in the oxidation of the cor pounds or ions named with an excess of K_2CrC This excess is determined by measuring the extin tion of the red colour formed by the reaction o-dianisidine with chromate in acid soln. Th oxidation products of the substances named do n interfere. *Procedure*—Potassium chromate sol (200 μg per ml) (2 ml) is transferred to a 100-r flask and the test soln. is added, followed by 3 r of 5N-H_2SO_4. The mixture is set aside to comple the reaction, and 0·5 ml of reagent soln. (0·5 g o-dianisidine dissolved in 50 ml of acetone ar diluted to 100 ml with water) is added. The sol is diluted to volume with water and the extinctic at 470 mμ is read between 5 and 30 min. after th addition of the reagent. Results are referred t an appropriate calibration curve. The procedu is suitable for concn. from 0·1 to 4 μg per ml.
H. M.

1488. **Rapid gravimetric determination of bismut as bismuth oxalate.** M. Vancea and M. Volușniu (Inst. Chem., Acad. R.P.R., Cluj, Romania). *Stu Cercet. Chim., Cluj*, 1959, **10** (2), 283–286.- Tervalent Bi is pptd. from dil. HNO_3 soln. wit 0·1N-oxalic acid. Since the ppt. is insol. in 2% HNO_3, the method can be used in the presence Ag, Pb, Cu, Fe, Co and Ni. The max. error f 5 mg of Bi per ml is ± 0·2%. H. SHER

1489. **The use of dithizone as internal indicato in precipitation titrimetry. I. Direct volumetri determination of bismuth as bismuth phosphat** M. Vancea and M. Volușniuc (Inst. Chem., Aca R.P.R., Cluj, Romania). *Stud. Cercet. Chim., Clu* 1959, **10** (2) 269–273.—A soln. containing 0·5% of Bi (10 to 20 ml) in 2% HNO_3 is titrate with 0·1N-KH_2PO_4 in the presence of 1 ml of 0·03% soln. of dithizone in $CHCl_3$. At the en point the orange Bi dithizonate changes throug grey to green.

III. Direct volumetric determination of bismut as bismuth arsenate. M. Vancea and M. Volușniu *Ibid.*, 1959, **10** (2), 279–281.—The same procedu is adopted with the use of 0·04M-Na_3AsO_4.

IV. Direct volumetric determination of bismut as bismuth oxalate. M. Vancea and M. Volușniu *Ibid.*, 1959, **10** (2), 287–290.—Approximately to 20 ml of a soln. of $Bi(NO_3)_3$ is treated with 5 r of acetic acid - Na acetate buffer soln. (pH 4· and 1 ml of a 0·03% soln. of dithizone in CHC The mixture is titrated with 0·1N-oxalic acid to green colour. H. SHER

1490. **Liquid-metal fuel-reactor experiment- spectrographic methods for bismuth analysis.** C. E Anderson and D. W. Brown (Babcock and Wilco Co., Research Center, Alliance, Ohio, U.S.A. *U.S. Atomic Energy Comm.*, Rep. BAW–109 1960, 36 pp.—Development of a rapid contr method for Mg and Zr that have been added a corrosion inhibitors to a circulating bismuth loop described. A rapid method of determining th amount of corrosion products in liquid bismuth

o reported. Magnesium and Zr can be quickly termined in 10% (w/v) bismuth in 8N-HNO_3 er the 100 to 600 p.p.m. range through the use a rotating disc technique. Coeff. of variation 2·4% for the Mg and 2·9% for the Zr determina- n were observed. It was found that Mg concn. to 10,000 p.p.m. had no effect on the Zr deter- nation. Some corrosion products were deter- ned by d.c. arc excitation on an ignited Bi_2O_3 mple. Adequate precision was attained and ere was good agreement between the spectro- aphic and chemical determinations of Fe and Modifications to the basic methods were efly investigated. Nucl. Sci. Abstr.

1491. **Extraction and flame-spectrophotometric termination of vanadium.** C. M. Stander (African etals Corp. Ltd., P.O. Box 66, Meyerton, Trans- al, S. Africa). *Anal. Chem.*, 1960, **32** (10), 96–1299.—After electrolysis at a mercury cathode remove Cu, Fe, Sn and Mo, V is extracted from . H_2SO_4 soln. with cupferron and ethyl acetate. e extract is diluted to 40 ml and aspirated into e burner of a Beckman DU flame spectrophoto- eter. The intensity of the emission of V at 529 550 mμ is referred to a standard curve. Toler- ce limits are quoted for 11 elements.

T. R. Andrew

1492. **Spectrophotometric determination of vana- um as molybdovanadic acid.** G. W. Wallace and G. Mellon (Purdue Univ., Lafayette, Ind., S.A.). *Anal. Chim. Acta*, 1960, **23** (4), 355–362 English).—At pH values between 1·5 and 6·0, reacts with MoO_4^{2-}, to form 5-molybdovanadic id (Mo to V ratio = 5; 1), with maximum forma- n at pH 3·0 to 3·5, and having a strong absorption 360 mμ (extinction 0·74 in 1-cm cell for 0·5 mg V per 50 ml). The interference of 31 ions has en studied; AsO_4^{3-}, Ba^{2+}, Bi^{3+}, citrate, $Cr_2O_7^{2-}$, $^{3+}$, GeO_4^{2-}, oxalate, Pb^{2+}, SiO_3^{2-} and tartrate ust be absent. Results on three standard steel mples, after prior removal of Fe, Cr and Si, re within 3% of the stated values.

T. R. Andrew

1493. **Separation of niobium, tantalum, titanium d zirconium from each other.** A. K. Majumdar d A. K. Mukherjee (Jadavpur Univ., Calcutta, dia). *Anal. Chim. Acta*, 1960, **23** (3), 246–247 English).—The acidified soln. containing Nb, , Ti and Zr is digested on a water bath with a rge excess of 5% pyrogallol soln. and set aside er-night. The ppt. of Nb and Ta pyrogallates filtered off, washed with weak pyrogallol soln., ied and ignited. The residue is fused with HSO_4, then dissolved, and the procedure is peated to effect complete separation of Nb and from Ti and Zr. The mixed oxides of Nb and are separated by the *N*-benzoyl-*N*-phenyl- droxylamine method (*Ibid.*, 1958, **19**, 23; 1959, , 245). The filtrates and washings are evaporated most to dryness and the organic matter is stroyed by wet oxidation with conc. HNO_3, ncn. H_2SO_4 and $HClO_4$. The soln. is cooled and uted, and the Zr is pptd. with salicylhydroxamic id in the presence of H_2O_2 (*Ibid.*, 1960, **22**, 25). ie Ti is pptd. from the filtrate with cupferron ter boiling to destroy H_2O_2 (Majumdar and owdhury, *Anal. Abstr.*, 1957, **4**, 450).

1494. **Volumetric determination of small amounts oxygen.** E. S. Boĭchinova, S. M. Efros and D. Nemirovskiĭ. *Trudy Leningr. Tekhnol. Inst. . Lensoveta*, 1959, (58), 31–35; *Ref. Zhur., Khim.*, 1960, (12), Abstr. No. 47,119.—The method is based on the oxidation of Ti^{3+} to Ti^{4+} and titration of unchanged Ti^{3+} with $FeCl_3$ soln. The rate of reaction between O and Ti^{3+} increases with increase of pH, but even in alkaline medium the rate is not sufficiently high for the O to be completely absorbed by a single passage of the gas through the absorbent soln. A closed system is used, comprising an absorbing vessel and two gas pipettes with pressure vessels. The gas sample is passed from the feed gas pipette through the absorbing vessel to the receiving gas pipette and then back to the feed pipette. Pure N is first passed through the system, then to the absorbing vessel are added 25 to 30 ml of standard $TiCl_3$ soln. in N-HCl and 10 to 15 ml of a satd. $CaCl_2$ soln., followed by excess of 3N-alkali (60 to 70 ml). The gas sample is passed through the absorbing vessel till absorption is complete (5 or 6 cycles); 3N-HCl is then added dropwise to dissolve the ppt. (a slight haze of TiO_2 may remain), 0·5 ml of 20% NH_4SCN soln. is added with the acid, and the unchanged Ti^{3+} are titrated with $FeCl_3$ soln. The method can be used for mixtures containing 0·1 to 1% of O. The error is $\not> 2\%$.

C. D. Kopkin

1495. **Method and apparatus for analysing a reactive gas [to determine its content of hydrogen sulphide].** Standard Oil Co. [Inventors: E. B. Offutt and L. V. Sorg]. Brit. Pat. 849,456; date appl. 24.5.57.—In a method for continuously and automatically determining the hydrogen sulphide concn. in air directly, without interference from other common sulphur-containing contaminants, *e.g.*, thiols and sulphur dioxide, a sample of the air is saturated with water and then applied under precise conditions to a moving transparent tape having a hydrogen sulphide-sensitive coating (film of gelatin impregnated with Pb acetate and Na acetate) which darkens in a quant. manner on exposure to hydrogen sulphide. The extent to which the light transmittancy of the film is reduced as it moves continuously through the exposure chamber is measured by means of a photo-electric cell located below the tape, the variations in the electrical response from the cell in accordance with the variations in the concn. of hydrogen sulphide in the gas sample being transmitted to a recorder.

J. M. Jacobs

1496. **Indirect colorimetric determination of sulphide and cyanide with thiofluorescein.** M. Wroński (Dept. Chem. Technol., Univ., Łódź, Poland). *Chem. Anal., Warsaw*, 1960, **5** (3), 457–460.—Alkaline soln. of thiofluorescein, decolorised with $AgNO_3$, become blue on the addition of H_2S or HCN. This phenomenon is used to determine H_2S (0·5 to 2 μg) and HCN (1 to 7 μg). The $AgNO_3$ - thiofluorescein reagent (**I**) is prepared by adding thiofluorescein soln. (5 mg in 50 ml of 0·1N-aq. NH_3) dropwise to 50 ml of 2×10^{-4}N-$AgNO_3$ until a permanent blue colour appears, then adding 20 ml of N-aq. NH_3 and diluting to 500 ml. A soln. of 2 g of As_2O_3 in 25 ml of N-NaOH treated with 5 g of Na_2CO_3 and 10 ml of conc. aq. NH_3 and diluted to 1 litre is used as a combined absorbent and diluent (soln. *A*). The determination is carried out by mixing 5 ml of sample with 5 ml of **I**, diluting to 12 to 25 ml with *A*, and measuring the extinction after 2 min. A blank and calibration standard are prepared at the same time. For the simultaneous determination of H_2S and HCN, after the extinction has been measured, a drop of

formaldehyde soln. is added to discharge the colour due to HCN, and the extinction due to H_2S alone is measured after 10 min. P. Brych

1497. **Spectrophotometric study of the Schiff reaction as applied to the quantitative determination of sulphur dioxide.** R. V. Nauman, P. W. West, F. Tron and G. C. Gaeke, jun. (Dept. Chem., Louisiana State Univ., Baton Rouge, U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1307–1311.—A study of the u.v. and visible spectra of the several products of the reactions between SO_2, formaldehyde, pararosaniline and HCl has been carried out. It is concluded that the purple reaction product is not a Schiff base, but a pararosaniline methylsulphonic acid. T. R. Andrew

1498. **Determination of sulphur dioxide and chlorate when present together.** I. E. Flis and T. A. Tumanova (Leningrad Technol. Inst.). *Zavod. Lab.*, 1960, **26** (8), 943–945.—The SO_2 is oxidised by a soln. of H_2O_2 of known concn. and the excess is determined by titration with a soln. of $KMnO_4$. The ClO_3^- are then determined by heating the soln. with excess of $FeSO_4$ soln., and titrating the excess with $KMnO_4$ soln.
G. S. Smith

1499. **New methods of control in bisulphite manufacture.** G. V. Zavarov (M. I. Kalinin Chernorechensk Chem. Works). *Zavod. Lab.*, 1960, **26** (8), 940–943.—To determine the relative amounts of sulphite and bisulphite in a soln. it is possible to use the soln. both as titrant and soln. being titrated. About 1 ml of the soln. containing $(NH_4)_2SO_3$ and NH_4HSO_3, of concn. corresponding to $\simeq$ 500 g of SO_2 per litre, is diluted to 100 ml with 0·05% EDTA (disodium salt) soln. (to prevent oxidation by the air), and 20 ml is poured into 10 ml of KIO_3 - KI soln. prepared by adding 2·5 g of KI and 1 ml of 0·01N-H_2SO_4 to 100 ml of a soln. of 25 g of KIO_3 in 1 litre. The soln. is treated with 1 g of $Na_2B_4O_7.10H_2O$ and the iodine liberated in the first reaction—

$$2NH_4HSO_3 + KIO_3 + KI = I_2 + K_2SO_4 + (NH_4)_2SO_4 + H_2O,$$

is titrated with the diluted sample soln., with starch as indicator, the reaction being—

$$(NH_4)_2SO_3 + I_2 + H_2O = (NH_4)_2SO_4 + 2HI.$$

G. S. Smith

1500. **Determination of sulphate ions by direct titration with lead nitrate using diphenylcarbazone indicator.** G. N. Nechiporenko. *Gidrokhim. Materialy*, 1959, **29**, 214–218; *Ref. Zhur., Khim.*, 1960, (15), Abstr. No. 61,098.—The procedure for the direct titration of SO_4^{2-} (*Anal. Abstr.*, 1958, **5**, 2008) is modified. Interference by heavy metals is suppressed by adsorption with a cation-exchange resin. With samples containing 0·01 and 0·025 mg of SO_4^{2-} the errors are $>$ 3% and 2%, respectively. With concn. of 25 to 100 mg of SO_4^{2-} per litre, micro-titration is recommended. Samples containing $<$ 10 mg of SO_4^{2-} per litre should first be treated with the cation-exchange resin and evaporated. The method may be used for the determination of SO_4^{2-} under field conditions.
K. R. Cook

1501. **Ultra-violet spectrophotometric determination of trace amounts of selenium with o-phenylenediamine.** Hisae Ariyoshi, Michio Kiniwa and Kyoji Tōei (Fac. Sci., Univ., Okayama, Japan). *Talanta*, 1960, **5** (2), 112–128 (in English).—Selenium reacts with *o*-phenylenediamine at pH 1·5 to 2·5 to give piaselenole, which can be extracted into toluene and determined by u.v. spectrophotometry. *Procedure*—To the sample soln. containing $>$ 30 μg of Se add a small amount of 80% formic acid and dilute to 50 ml with water. Adjust the pH to 1·5 to 2·5, add 0·2% *o*-phenylenediamine soln. (2 ml) and set aside for 2 hr. Extract with toluene (10 ml), centrifuge and measure the extinction of the toluene layer at 335 mμ; Al, NH_4^+, Sb, As, Ba, Cd, Ca, Cr, Co, Cu, Pb, Mg, Mn, Ni, K, Na, NO_3^-, SO_4^{2-}, PO_4^{3-} and tartrate do not interfere but Bi, Sn and I^- must be absent. Interference from Fe^{III} can be prevented by the addition of EDTA. W. T. Carter

1502. **Extraction of chromium(III) with thenoyltrifluoroacetone. Direct spectrophotometric determination in the organic phase.** S. K. Majumdar and Anil K. De (Jadavpur Univ., Calcutta, India). *Anal. Chem.*, 1960, **32** (10), 1337–1339.—At pH $\simeq$ 6, Cr^{3+} react with 2-thenoyltrifluoroacetone in benzene to form an orange benzene-sol. chelate. The extraction is 100% at pH 5·75, 70% at pH 6·5 and 50% at pH 6·7, after shaking for 15 min. Of 13 ions examined, Ag^+ and Al^{3+} may be tolerated at levels up to twice that of the Cr^{3+}. The extinction is measured at 430 mμ. T. R. Andrew

1503. **Determination of chromate in the presence of arsenate or permanganate.** Á. Kellner, L. G. Molnár and L. Szekeres (Chem. Dept., Agric. Univ., Budapest). *Magyar Kém. Lapja*, 1960, **15** (9), 426–427.—Volumetric methods are described. *Procedure for the determination of chromate in the presence of arsenate*—The soln. is treated with 0·1N-KBr in H_2SO_4 medium. The reduction of chromate is complete in $\simeq$ 15 min.; KI and starch soln. are then added and the mixture is titrated with standard arsenite soln. *Procedure for the determination of chromate in the presence of permanganate*—The reduction of permanganate to MnO_2 is carried out with Na_2CO_3 and H_2O_2. The excess of H_2O_2 is destroyed by heating on a water bath, the soln. is acidified with H_2SO_4 and KI is added. The liberated iodine is titrated with standard $Na_2S_2O_3$ soln. The procedure is rapid, but correction factors must be applied. B. Vaszko

1504. **Process and apparatus for potentiometrically measuring and reducing chromate ions in aqueous solution.** Deutsche Gold- und Silber-Scheideanstalt, vormals Roessler. Brit. Pat. 853,326; date appl. 9.5.58. Germany, date app. 25.5.57.—The potentiometric determination of extremely small quantities of chromate in waste liquors can be carried out with the aid of a measuring electrode, if necessary in the absence of quinhydrone, and the addition of the usual reducing agents, *e.g.*, $NaHSO_3$ or SO_2, is thereby controlled. The measurement is advantageously carried out in a small partial stream, which is branched off from the solution to be treated and fed to a measuring station, from which it is returned again to the main stream. J. M. Jacobs

1505. **Compounds of quinquevalent molybdenum with 8-hydroxyquinoline and their analytical use.** A. I. Busev and Chzhan Fan (Lomonosov Univ., Moscow). *Zhur. Anal. Khim.*, 1960, **15** (4), 455–462.—Quinquevalent Mo forms two compounds with 8-hydroxyquinoline: a greenish-yellow ppt. produced at room temp. and a black ppt. produced in a boiling soln. The composition of the greenish-yellow ppt. corresponds approx. to th

ormula $H_2Mo_4O_{11}(C_9H_7NO)_7.11H_2O$ and that of he black ppt. corresponds exactly to the formula $Mo_2O_3(C_9H_6NO)_4.H_2O$. The black compound of Mo^{5+} can be used for the photometric determination of small amounts of Mo (0·1 μg per ml) in a 0% acetone medium. The method gives accurate esults and Ca, Mg, Cd, Ni, Zn and small amounts f Fe^{3+} and Cr^{3+} do not interfere. *Procedure*—dd conc. HCl (4 ml) to a neutral or slightly acid oln. containing $\gtrsim$ 0·8 mg of Mo^{6+}. Dilute the oln. to 13 to 15 ml, heat it on a boiling-water ath and slowly add an aq. reducing soln. (0·1 g f hydrazine dihydrochloride per ml) (5 to 7 ml); ontinue heating for 10 to 20 min. Add 3% 8-ydroxyquinoline soln. in dil. acetic acid (4 ml) to he hot soln. while stirring and then aq. NH_3 oln. until a ppt. appears. Then add acetate uffer soln. (2 ml) at pH 4 and heat for 10 to 15 in. on a boiling-water bath. Cool the mixture o room temp., add acetone (25 ml) and dilute the oln. to 50 ml. Stir well and measure the extinc-ion at 530 mμ. K. R. Cook

1506. **Determination of molybdenum in nitrilo-riacetic acid medium by derivative polarography. Application to solutions of mixed thorium - uranium oxides.** D. L. Manning, R. G. Ball and O. Menis (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1247–1250.—The effect of a umber of complexing agents on the $E_{\frac{1}{2}}$ of U^{VI} nd Mo^{V} is reported. Maximum separation $E_{\frac{1}{2}}$ = − 0·23 V for U^{VI} and − 0·48 V and − 0·64 V for Mo) is obtained in a medium 0·15M n nitrilotriacetic acid at pH 3·0. Interferences have been studied at the level of 70 μg of Mo; 100 μg of Cr, Cu, Ti and Ni, 500 μg of Fe, 1 mg of U or 25 mg of Th may be present in 10 ml without causing an error greater than that implicit in the method (coeff. of variation $\simeq$ 6%).

T. R. Andrew

1507. **Determination of molybdenum and tungsten by an acidimetric method.** A. I. Lazarev (Akmolinsk Agric. Inst.). *Zavod. Lab.*, 1960, **26** (8), 935–938.—The method is based on potentiometric titration of the soln. in a vessel of antimony, which serves as the working electrode, with a reference silver - silver chloride electrode which serves also as a stirrer. G. S. Smith

1508. **Chemical - spectrographic determination of small amounts of molybdenum and tungsten in minerals.** L. I. Pavlenko (Vernadskiĭ Inst. of Geochem. and Anal. Chem., Acad. Sci., Moscow). *Zhur. Anal. Khim.*, 1960, **15** (4), 463–466.—When the concn. of Mo and W is $< 10^{-4}$ %, an enrichment is carried out before spectrographic determination with a d.c. arc. The sample (10 mg), a mixture of 2 mg of the residue obtained after chemical enrichment of the mineral by means of organic co-precipitants with silicate base (18 mg), is completely volatilised from the crater of a carbon electrode (depth 2·5 mm and diam. 3·5 mm). The slit width is 0·02 mm, the current 10 amp., and the exposure time 2·5 min. The spectra are photographed with type-II plates and sensitivity 8 or 11 units (GOST) for Mo, and 22 units (GOST) for W; the lines used are 3170·35 A (Mo) and 4294·6 A (W). Calibration curves are constructed by using the triple-standard method for log R *vs.* log C, where R is the relative intensity of the test line in comparison with the background and C is the concn. (%) of the test element. To prepare standards, MoO_3 and WO_3 are introduced into the base. When the mineral contains $> 10^{-4}$ % of Mo and W, the determination is carried out by direct spectrographic determination. The sensitivities of the chemical - spectrographic determination of Mo and W are 2×10^{-6} % and 3×10^{-5} %, respectively. K. R. Cook

1509. **Spectrophotometric determination of molybdenum in alloys in the presence of tungsten, silicon and aluminium.** I. P. Kharlamov, P. Ya. Yakovlev and M. I. Lykova. *Zavod. Lab.*, 1960, **26** (8), 933–934.—The sample (0·1 to 0·5 g), together with 0·1 g of Fe if the alloy is based on Ni, Co or Cr, is dissolved in 50 ml of conc. HCl followed by 3 to 5 ml of conc. HNO_3 for oxidation, and the cooled soln. is treated with 10 ml of conc. H_2SO_4 and evaporated twice to fuming. The cooled residue is dissolved in 80 ml of water and the soln. is poured gradually into 100 ml of boiling 25% NaOH soln. After dilution of the cooled soln. to 500 ml it is set aside for 1 to 2 hr. An aliquot (250 ml) of the filtered soln. is treated with H_2SO_4 (1:4) until the pH is between 6 and 8, and then diluted to 500 ml. The extinction of the soln. is measured at 230 mμ, and the content of Mo is calculated by use of a calibration curve and a blank test. The method gave satisfactory results over the range 1 to 10% of Mo. G. S. Smith

1510. **Determination of tungsten and molybdenum without their separation.** E. Lassner and R. Scharf (Metallwerk Plansee A.-G., Reutte, Tirol, Austria). *Chemist Analyst*, 1960, **49** (3), 68–69.—Tungstate and molybdate are determined together by pptn. titration with Pb soln. and Mo is determined separately by titration with EDTA. *Procedures*: (*i*) *For W and Mo*—Acidify the soln. (approx. 0·8 millimole of W and Mo) with HNO_3 (1:1) and boil. Buffer at pH 6·8 with hexamine. Add 5 to 10 drops of 0·1% aq. 4-(2-pyridylazo)-resorcinol soln. and titrate the boiling soln. (300 to 400 ml) at a rate of 1 drop per sec. with 0·1M-$Pb(NO_3)_2$ to a permanent red colour. (*ii*) *For Mo* (0·05 to 0·2 *millimole*)—Add a known vol. of 0·05M-EDTA (disodium salt), tartaric acid (5 g), hydrazine sulphate (2 to 3 g) and H_2SO_4 (1:1) (2 ml). Dilute to $\simeq$ 170 ml, boil for 5 min. and adjust the pH to 4 to 5 with aq. NH_3. Add 80 ml of methanol and 5 to 8 drops of 0·1% ethanolic 1-(2-pyridylazo)-2-naphthol soln. Titrate with 0·05M-$CuSO_4$ and finally restore the yellow colour by back-titration with EDTA. For 5 to 50 mg of Mo, a photometric determination at 450 mμ is described.

P. D. Parr-Richard

1511. **Interference of iron in the determination of 12-tungstophosphoric acid with tri-n-butylamine.** C. C. Miller and D. H. Thow (Chem. Dept., Univ., Edinburgh, Scotland). *Talanta*, 1960, **5** (2), 128–130 (in English).—The interference of Fe^{III} in this method (*Anal. Abstr.*, 1960, **7**, 972) is due to the pptn. of $[Fe(H_2O)(C_{12}H_{27}N)_5]PW_{12}O_{40}.nH_2O$. It can be prevented by reducing the Fe^{III} to Fe^{II} in a silver reductor, followed by pptn. of the complex from 2N-HCl. W. T. Carter

1512. **Procedure and problems in the fluorimetric determination of uranium.** T. Schönfeld, M. ElGarhy, C. Friedmann and J. Veselsky (Anorg. u. Phys. Chem. Inst., Univ., Wien, Austria). *Mikrochim. Acta*, 1960, (5–6), 883–897 (in German).—Uranium is determined in rocks, coal ash or leachings from coal ash by separating it from 8% HNO_3 soln. with ethyl acetate and transferring it

to a fluoride pellet for fluorescence measurement. Errors due to the properties of the pellet, to sample-vol. measurement and to the photometer are evaluated. Quenching by interfering elements is eliminated by solvent extraction followed by the method of standard addition. *Procedure*—For coal ash, evaporate the sample several times with HF and then with HNO_3 to give a soln. 8% in HNO_3. For alkali carbonate extracts, add HNO_3 to expel CO_2 and adjust to 8%. For H_2SO_4 extracts, precipitate SO_4^{2-} as $BaSO_4$; traces of Fe^{3+} or Al^{3+} prevent adsorption of U. Mix a 2-ml aliquot of the soln. with 10 ml of saturated $Al(NO_3)_3.8H_2O$ soln. (0·9 g per ml) and extract U with ethyl acetate (10 ml). Add 0·2 ml of extract to a pellet (NaF - LiF) (49 : 1) weighing 0·4 g, dry under a i.r. lamp and melt over a burner. Measure the fluorescence of the solidified melt, add a known amount of U, re-melt and measure again. For $< 20 \times 10^{-9}$ g of U per g of sample, concentrate the solvent extract and decant from the pptd. $Al(NO_3)_3$. P. D. PARR-RICHARD

1513. **Direct determination of uranium in concentrates by spectrophotometric titration with ferric sulphate.** T. M. Florence (Australian Atomic Energy Comm., Lucas Heights, N.S.W.). *Anal. Chim. Acta*, 1960, **23** (3), 282–288 (in English).—Uranium may be determined by reduction to U^{IV} and then titration with $(NH_4)_2SO_4.Fe_2(SO_4)_3$ soln.; Cu, Mo and V interfere, and their maximum permissible concn. are discussed. The sample (50 to 100 mg of U_3O_8) is dissolved in $HClO_4$, diluted and acidified further with H_2SO_4 and diluted to 25 ml. A portion is transferred to a special reduction vessel (illustrated), and de-gassed with N. Bismuth amalgam is added and the vessel is shaken vigorously for 5 to 10 min. The special titration cell (illustrated) is placed in the spectrophotometer, 5 ml of water is added and the instrument is set at 650 mμ. Nitrogen is passed through, 5 ml of reduced soln. is added to the cell and the instrument is set to zero. The gas flow is then stopped and the extinction is measured. Nitrogen is passed through again, a little titrant (0·1N) is added, the gas flow is stopped and the extinction is again measured. This procedure is repeated to obtain a titration curve. H. M.

1514. **Determination of total uranium and uranium-235 in aluminium - uranium fuel elements.** W. L. Delvin, H. E. Palmer and U. L. Upson (General Electric Co., Hanford Atomic Products Operation, Richland, Wash., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. HW–57464, 1958. Decl. Apr. 1960. 22 pp.—Uranium - aluminium alloy fuel elements containing 3 to 5% of enriched U were analysed both for total U content and for the degree of enrichment (≃ 93% of ^{235}U). Each fuel element was reduced to wafers and turnings which were dissolved in HCl and Br. For the determination of total U, the U was reduced in a Jones reductor and re-oxidised with excess of standard $Ce(SO_4)_2$ soln., with subsequent back-titration of the excess of ceric ion with $FeSO_4$ soln. A correction for iron interference was necessary. The precision of this method is ± 0·3% at the 95% confidence level. For isotopic analysis, the uranium soln. were γ-counted in the 0·174 to 0·194-MeV interval, without chemical separation, and evaluated relative to mass-spectrometer-calibrated standards. Corrections for both U and $AlCl_3$ concn. were applied. Over-all counting precision is ± 0·4% or better. The reported isotopic concn. were derived from the determination of total U and the γ-counting results, and are precise to ± 0·5%. NUCL. SCI. ABSTR.

1515. **Extraction of irradiated uranium by ketones in acid and acid-deficient solutions.** V. Veselý, H. Beranová and J. Malý (Inst. for Nuclear Research, Czech. Acad. Sci., Prague). *Coll. Czech. Chem. Commun.*, 1960, **25** (10), 2622–2629 (in German).—The extraction of uranium and its fission products from soln. 0·25M in $UO_2(NO_3)_2$ and 5M in NH_4NO_3 by a series of alkyl methyl ketones was found to vary with the concentration of free HNO_3 in nitrate soln. In the case of uranium this dependence is attributed to the formation of basic nitrates of the type $[UO_2(OUO_2)n].(NO_3)_2$, which, unlike $UO_2(NO_3)_2$, are not soluble in ketones. The partition coeff. of uranium decreases with decreasing acid concn.; its logarithm increases linearly with the logarithm of the carbonyl content of the ketone. The partition coeff. of the fission products varies similarly. The efficiency of the separation of uranium from its fission products can be expressed by the decontamination factor (DF), defined as the ratio $(A_v/C_v)/(A_o/C_o)$, where A_v and A_o are the activities of 1 g of aq. phase and 1 g of organic phase, respectively, and C_v and C_o are the corresponding concn. (w/w) of U. The DF is highest in soln. with a small acid deficiency (— 0·05M in free HNO_3) and lowest in neutral soln. and in those with a high acid deficiency; in strongly acid soln. it is greater than in neutral soln. In both acid and acid-deficient soln. the DF increases with increasing number of carbon atoms in the ketone. It is concluded that the separation of U from its fission products is best carried out by extraction with isobutyl methyl ketone from strongly acid soln. (≃ 3M in free HNO_3) or those with a small acid deficiency. R. M. ROWLEY

1516. **The mechanism of the extraction of uranyl nitrate by tributyl phosphate. II. Infra-red study.** K. Nukada, K. Naito and U. Maeda (Govt. Chem. Ind. Res. Inst. of Tokyo, Shibuya). *Bull. Chem. Soc. Japan*, 1960, **33** (7), 894–898.—The mechanism of the extraction of uranium by tri-n-butyl phosphate (TBP) is studied by means of i.r. spectra from Perkin - Elmer Model 12C and 321 spectrometers with rock-salt prisms. The systems TBP - water, TBP - nitric acid, TBP - uranyl nitrate, and TBP - uranyl nitrate - nitric acid are presented. It is concluded that the competitive extraction of both uranyl nitrate and nitric acid takes place during the process. E. M. MAYES

1517. **Application of gas - liquid chromatography to the analysis of corrosive gases. I. Development of apparatus and technique for separation of mixtures of chlorine trifluoride, hydrogen fluoride and uranium hexafluoride.** G. Iveson (Tech. Dept., U.K.A.E.A. Production Group, Capenhurst). U.K.A.E.A. Report PG82(CA), 1960. 23 pp.—Mixtures of UF_6, HF and ClF_3 may be separated with N or He as the carrier gas, a packing of "Kel-F" moulding powder [poly(chlorotrifluoroethylene)] and "Kel-F 40" polymer oil in a nickel column at 60°, and katharometer detection. The response was linear with concn. for ClF_3 and UF_6, but was non-linear for HF, the response increasing rapidly with increase in HF concn. The method is suitable for up to 25% by vol. of HF, 15% by vol. of ClF_3 and 15% by vol. of UF_6 in a sample vol. of ≃ 20 ml at s.t.p. T. R. ANDREW

1518. **Fluorimetric determination of uranium in rconium and hafnium.** P. A. Vozzella, A. S. owell, R. H. Gale and J. E. Kelly (Nuclear Div., mbustion Engineering Inc., Windsor, Conn., .S.A.). *Anal. Chem.*, 1960, **32** (11), 1430–1433.—ranium (≃ 1 p.p.m.) in Zircaloy or in pickling ln. is determined fluorimetrically in a Jarrell-Ash strument after extraction from HNO_3 soln. into hyl acetate. Results are accurate to within 5%. *Procedure*—Dissolve the Zircaloy (0·5 g) water (10 ml) by adding 48% HF (2 ml), 30% $_2O_2$ (9 drops) and conc. HNO_3 (5 ml). Evaporate dryness, dissolve the residue in water, add nc. HNO_3 (1 ml), and dilute to 20 ml. To 2 ml this soln. add satd. aq. $Al(NO_3)_3$ (15 ml) and lid $Al(NO_3)_3.9H_2O$ (0·5 g), and extract for 2 to min. with ethyl acetate (5 ml). Pipette 0·4 ml the organic phase on to four pellets (0·4 g each) a flux of 98% of NaF and 2% of LiF. Fuse, ol, and measure the fluorescence. Determine the ntent of U by comparison with standards.

J. P. Stern

1519. **The determination of some impurities in igh-purity uranium. The use of anion exchangers or the separations.** D. B. Stevančević (Inst. of Nucl. Sci. "Boris Kidrich," Beograd-Vinca). *Bull. oc. Chim., Belgrade*, 1959, **23–24** (3–4), 185–190.—he determination of μg amounts of Cu, Cd, Zn, Ti, Co, Fe, Mn and Al in high-purity uranium is ffected by isolating the total impurities from dil. H_2SO_4 (pH 1·5) on Dowex–1 resin, followed by lution with 6N-HCl (for Ni, Al and Mn), 0·5N-ICl (for Co, Cu and Fe) and 0·0005N-HCl (for Cd nd Zn). A reproducibility of 5·2 to 15·2% for ach element is attained. The method is applied o the analysis of a sample of uranium peroxide.

M. G. Seaman

1520. **Determination of trace amounts of cadmium n uranyl nitrate with dithizone.** A. Gołkowska Inst. for Nucl. Res., Polish Acad. Sci., Warsaw). *Chem. Anal., Warsaw*, 1960, **5** (3), 389–394.—*Procedure*—Dissolve 0·2 g of uranyl nitrate in vater, transfer to a 50-ml separating-funnel, add ml of 17% ammonium carbonate soln., mix and dd 1 ml of conc. aq. NH_3. Extract with 10-ml portions of dithizone soln. (0·002% in CCl_4) till he extracting layer is green, then extract with a -ml portion. Wash the combined extracts with 0 ml of water, then shake them with 10 ml and ml of 0·01N-HCl. Wash the acid extract with ml of dithizone soln. and then with 5 ml of CCl_4. To the acid extract add 5 ml of 10% citric cid soln., 5 ml of dithizone soln. (0·005% in CCl_4) and 5 ml of 20% NaOH soln. Shake for 1 o 2 min. and filter through cotton wool into a -cm cell. Measure the extinction at 520 mμ gainst a blank and refer the result to a calibration raph. The dithizone - Cd complex showed no hange in extinction during 2 hr. The accuracy vas not affected by a fivefold excess of Ag^+, Bi^{2+}, Cu^{2+}, Hg^+, Ni^{2+}, Fe^{3+}, Zn^{2+}, Pb^{2+} or Co^{2+} indi-vidually, nor by the combined addition of a 2·5-old excess each of Zn^{2+}, Pb^{2+}, Bi^{2+}, Fe^{3+}, Cu^{2+} nd Co^{2+}. The errors were ≯ 5% in the range to 10 μg of Cd, increasing to 10% in the 0·1 to 0·5-μg range. The sensitivity can be increased to 0·1 μg of Cd per g of uranyl nitrate by taking up o 1 g of sample and proportionately increasing he amounts of ammonium carbonate and aq. NH_3 oln.

P. Brych

1521. **Determination of aluminium in pure uranium and in aluminium - uranium alloys (8-hydroxyquinoline spectrophotometric procedure).** C. O. Granger (U.K.A.E.A., Development and Engineering Group, Culcheth). U.K.A.E.A. Report DEG 219(C), 1960. 17 pp.—The development of the method is described. The sample, containing up to 5 g of U, is dissolved in HCl and H_2O_2. Thioglycollic acid, EDTA, carbonate buffer soln., cyanide and 8-hydroxyquinoline (**I**) are added; after 1 hr. the Al is extracted as its complex with **I** into $CHCl_3$. The extinction of the soln. at 390 mμ is measured and the content of Al is derived from a calibration graph obtained by using known amounts of Al. If the alloy contains Ti, V or Zr, these are extracted as complexes with *N*-benzoyl-*N*-phenylhydroxylamine before the normal procedure is applied. Full details are appended. The standard deviation for contents of Al up to 50 μg is ± 0·3 μg.

G. J. Hunter

1522. **Determination of free acid in plutonium solutions.** J. L. Pflug and F. J. Miner (Rocky Flats Plant, Dow Chemical Co., Denver, Colo., U.S.A.). *Anal. Chim. Acta*, 1960, **23** (4), 362–367 (in English).—An investigation was made of the use of K oxalate, K tartrate and Na citrate as complexing agents to prevent hydrolysis and pptn. of Pu during potentiometric titration of free acidity. The use of Na citrate at pH 8·0 to 9·0 is recommended; after addition of the sample soln. the soln. is titrated potentiometrically to the original pH.

T. R. Andrew

1523. **Analysis of halogen mixtures by micro-diffusion in combination with isotope-dilution technique.** K. J. Öbrink and M. Ulfendahl (Univ. Uppsala, Sweden). *Acta Soc. Med. Upsaliensis*, 1959, **64**, 384–391.—A modification of the micro-diffusion method of Conway is described for the separate determination of Cl, Br and I. Analysis of some mixtures of two halogens is possible, but determination of Cl or I in bromine is not possible. By the conventional procedure for microdiffusion analysis, only Br can be determined in a mixture of all three halogens. However, by combining microdiffusion analysis and the isotope-dilution technique it is possible to determine all three halogens in mixtures.

Chem. Abstr.

1524. **Determination of fluorine by neutron activation.** O. U. Anders (Radiochem. Lab., Dow Chemical Co., Midland, Mich., U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1368–1369.—The method described is based on the ^{19}F $(n.\alpha)^{16}N$ reaction and can be used to determine 100 p.p.m. of F in a 1-g sample. To improve the sensitivity and statistical certainty of the method, a fast-transfer shuttle-rabbit system permits the use of multi-activation runs with cumulative counting.

K. A. Proctor

1525. **Colour reaction for fluoride ion.** H. Weisz (Coates Chem. Lab., Louisiana State Univ., Baton Rouge, U.S.A.). *Mikrochim. Acta*, 1960, (5–6), 703–705 (in German).—Use is made of the high stability of FeF_3 which is formed on addition of F^- to ferric rhodizonate in the presence of $AgNO_3$, the liberated rhodizonic acid yielding a blue-black Ag rhodizonate. Marked reaction is observed with 15 μg of F^- in 0·05 ml. Oxalate, S^{2-} and Ca^{2+} interfere; CrO_4^{2-}, PO_4^{3-}, salicylate and sulphosalicylate reduce the sensitivity. *Procedure*—To a drop of the neutral sample on a spot-plate

or on filter-paper add a drop of reagent [prepared by mixing 1% $(NH_4)_2SO_4.Fe_2(SO_4)_3$ soln. (2 ml) with 1% $AgNO_3$ soln. (2 ml), adding Na rhodizonate until a ppt. forms, filtering, adding acetic acid (1 drop) and diluting to 10 ml with 1% $AgNO_3$ soln.]. A blue - black ppt. appearing within 1 min. indicates F^-. J. P. Stern

1526. **Determination of ionisable fluorides in gases.** Mine Safety Appliances Co. Brit. Pat. 851,270; date appl. 1.4.59. U.S.A., date appl. 4.4.58.—When a gas, *e.g.*, air, containing HF or other ionisable fluoride is brought into contact with an inert porous material (filter-paper) carrying, as reagent, a freshly prepared suspension in an aq. HCl medium (containing sugar or glycerol) of the reaction product of 4-arsono-4′-dimethylamino-azobenzene and an excess of $ZrOCl_2.8H_2O$ over a 1:1 mol. ratio, the reagent assumes a brown to deep-pink colour, the exact hue being dependent on the concn. of the fluoride in the gas being tested. With a vol. of 1 litre of gas as the sample, the reagent has a sensitivity of $\simeq 0{\cdot}2$ p.p.m. of hydrogen fluoride. J. M. Jacobs

1527. **Analysis of chlorine cell gas by gas chromatography.** E. E. Neely (Columbia-Southern Chem. Corp., Corpus Christi, Tex., U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1382–1383.—The method described has been developed specifically for the analysis of chlorine cell gas, but is adaptable to any sample that requires a two-column separation. The gases are passed first through a 12-ft. column of 30% (w/w) Fluorolube grease (Type LG–160) on 30 to 50-mesh Chromosorb, and any gases unresolved are then passed through an 8-ft. Molecular Sieve column (Type 13X), 30 to 50 mesh. A thermal conductivity detector is used. C. B. Baines

1528. **Volumetric assay of ammonium perchlorate.** E. A. Burns and R. F. Muraca (Jet Propulsion Lab., Inst. of Technol., Pasadena, Calif., U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1316–1318.—*Procedure*—Dissolve 1·0 g in de-aerated water and dilute to 250 ml. Transfer 25 ml to a 500-ml conical flask containing 50 ml of HCl, and add 1 to 2 g of $NaHCO_3$ and 50 ml of 0·3N-$TiCl_3$. Add rapidly 1 ml of OsO_4 soln. (0·008M in 0·1N-H_2SO_4). Close the flask with a stopper carrying a tube leading to a flask containing 100 ml of 12N-HCl and connected to a third flask containing 100 ml of satd. $NaHCO_3$ soln. Boil the test soln. for 10 min. and cool to room temp. in an ice bath ($NaHCO_3$ soln. should be drawn back into the second flask so as to evolve CO_2). Titrate with 0·33N-$(NH_4)_2SO_4.Fe_2(SO_4)_3$, with NH_4SCN as indicator. Standard deviations of 0·11 to 0·19 are quoted for 4 determinations on each of 3 samples of 99·3 to 99·8% purity. All soln. and the water used must be de-aerated by bubbling with N for 30 min. T. R. Andrew

1529. **Impregnation method for the micro-determination of bromide and iodide.** A. J. Hłyńczak, J. S. Knypl and R. Antoszewski (Dept. of Physiol., Acad., Łódź, Poland). *Chem. Anal., Warsaw*, 1960, **5** (3), 407–411.—Micro amounts (1 to 30 μg) of bromide and iodide can be determined by placing a drop of the test soln. on filter-paper impregnated with Ag_3AsO_4, developing, fixing and measuring the area of the resulting spot. Mixtures give a double spot, with iodide at the centre, which permits the simultaneous determination of the two halides. Errors are $< 5\%$. The method is suitable for the determinations of the specific radioactivity of samples containing ^{131}I, ^{82}Br and ^{80}Br. P. Brych

1530. **Detection of iodine compounds by paper chromatography.** J. M. Calvo and L. Bascur (School of Med., Univ., Santiago, Chile). *Nature*, 1960, **188**, 141–142.—The rapid fading of chromatograms caused by traces of iodine in the air when detecting inorganic and organic iodine-containing compounds by the $Ce(SO_4)_2$ - As_2O_3 reagent (Kolthoff and Sandell, *J. Amer. Chem. Soc.*, 1934, **56**, 1426) can be obviated by exposing the paper, immediately after development, to the vapour of 50% phenol soln. before drying in air. The iodine-containing compound is thereby fixed as a white spot on a dark-brown background. W. J. Baker

1531. **Determination of periodate by the formaldehyde formed on oxidation of ethanediol.** L. Maros, I. Perl-Molnár and E. Schulek. *Magyar Kém. Foly.*, 1960, **66** (9), 342–344.—This is a version (in Hungarian) of the paper published (in German) in *Acta Chim. Acad. Sci. Hung.*, 1960, **22** (4), 475 (*cf. Anal. Abstr.*, 1961, **8**, 562). B. Vaszko

1532. **Arsenate - iodimetric determination of manganese.** G. B. Shakhtakhtinskiĭ and R. A. Melikova. *Azerbaidzh. Khim. Zhur.*, 1959, (5), 63–69; *Ref. Zhur., Khim.*, 1960, (16), Abstr. No. 65,115.—The method is based on the pptn. of Mn^{2+} by an excess of AsO_4^{3-}, followed by the dissolution of the ppt. in acid and iodimetric titration of the liberated H_3AsO_4. K. R. Cook

1533. **Spectrophotometric determination of technetium(VII) with thioglycollic acid.** F. J. Miller and P. F. Thomason (Anal. Chem. Div., Nat. Lab., Oak Ridge, Tenn., U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1429–1430.—The complex of Tc (4 to 20 μg) with thioglycollic acid (**I**) formed at pH 8·0 is determined absorptiometrically at 655 mμ. A tenfold excess of Cl^-, F^-, Br^-, I^-, PO_4^{3-}, SO_4^{2-} or tungstate does not interfere, but $Cr_2O_7^{2-}$, molybdate and ruthenate interfere. No accuracy is stated, but the reproducibility is within $\pm$ 2%. *Procedure*—To the soln. of $KTcO_4$, containing 10 to 200 μg of Tc, add M-Na acetate (1 ml) and 10% (v/v) **I** soln. (pH 8·0 $\pm$ 0·2) (1 ml). Heat at 100° for 15 min., dilute to 5 ml with water, and measure the extinction at 655 mμ against a blank. J. P. Stern

1534. **Chemical analysis on the microgram scale. VI. The ultra-micro gravimetric determination of technetium and rhenium.** F. Jasim, R. J. Magee and C. L. Wilson (Chem. Dept., Univ., Belfast, N. Ireland). *Mikrochim. Acta*, 1960, (5–6), 721–728 (in English).—The scheme previously described (*Talanta*, 1960, **4**, 17) for the separation of Tc, Re, Mn, Ru and Mo is adapted to ultra-micro gravimetry. The Tc and Re are pptd. and weighed as tetraphenylarsonium pertechnetate and perrhenate, respectively; 2 to 3 μg of Tc and Re can thus be determined with a standard deviation of $\pm$ 0·08 μg. J. P. Stern

1535. **Elimination of the effect of molybdenum on the colorimetric determination of rhenium.** B. S. Tsȳvina and N. K. Davidovich (State Sci. Res. and Development Inst. of the Rare Metals Industry). *Zavod. Lab.*, 1960, **26** (8), 930–932.—*Thiourea method* (*for contents of Re between* 0·001 *and* 0·1%)—The sample (0·3 to 2 g) is mixed with 3 to 4 g of CaO and heated at 650° to 700° for 2 hr.

ie mass is boiled with 150 ml of water until the l. is reduced to 100 ml (20 to 25 min.). The in. is filtered through a Buchner funnel and the sol. matter is washed with hot water. The rate is treated with 5 ml of 10% $BaCl_2$ soln. and iled until the vol. has been reduced to 80 ml if the content of Re is in the lower part of the nge, to 35 ml. The ppt. is filtered off and washed th hot water. The filtrate is diluted to a suitable l. and an aliquot (10 ml) is treated with 5 ml of nc. HCl, 2·5 ml of 10% thiourea soln. and 1 ml 20% $SnCl_2$ soln., and then diluted to 25 ml. ter 30 min. the extinction is measured with a olet filter. *Thiocyanate method* (*for contents of between* 0·0002 *and* 0·001%)—The sample (2 g) heated with CaO (4 g) as described above and e mass is extracted with hot water and the soln. filtered. The filtrate is treated with 5 to 6 ml 10% $BaCl_2$ soln. and evaporated to 35 to 40 ml. ie soln. is again filtered, and an aliquot (25 ml) evaporated to between 7 and 8 ml. The soln. treated with 10 ml of conc. HCl and the cooled ıd diluted soln. (20 ml) is treated with 1 ml of a ln. containing 10 mg of Fe as $FeCl_3$, 2 ml of •% NH_4SCN soln. and 1 ml of 35% $SnCl_2$ soln. ie soln. is diluted to 25 ml and the extinction is easured after 30 min. with a violet filter.

G. S. Smith

1536. **Specific spot test for iron.** K. Lal Mallik ıd Buddhadev Sen (Coates Chem. Lab., Louisiana tate Univ., Baton Rouge). *Anal. Chim. Acta*, 960, **23** (3), 225–226 (in English).—Two methods re described. *Procedure* (*i*)—One drop of test ln. is mixed on a spot-plate with a few crystals a reducing agent (*e.g.*, ascorbic acid, hydroxy-mmonium chloride or $Na_2S_2O_5$), followed by 2 rops of reagent soln. (1% of phenyl 2-pyridyl etoxime in 95% ethanol). The soln. is made kaline with 0·5M-aq. Na_2CO_3 or 6M-aq. NH_3. violet colour indicates the presence of Fe. *Pro-dure* (*ii*)—One drop of test soln. is placed on Vhatman No. 40 paper, followed by two drops 10% aq. ascorbic acid soln. and two drops of agent soln. The spot is exposed to NH_3 vapour. violet coloration indicates the presence of Fe. he only ions that interfere are Cu^{2+} and Co^{3+} n amounts > 10 times that of the Fe). Cyanide hibits the test. The limit of identification is 05 μg and the limit of dilution is 1 in 10^6.

H. M.

1537. **Test for iron by automatic concentration a small area of reagent.** F. L. Hahn. *Mikrochim. cta*, 1960, (5–6), 675–677 (in German).—The test or Fe^{3+} has a limiting concn. of 0·06 μg per ml nd requires highly pure Fe-free NH_4SCN. *Pro-dure*—Prepare the reagent as follows: to 5% aq. ccinic acid (20 ml) at pH 4·2 add NH_4SCN (25 g) nd then $K_4Fe(CN)_6$ (0·4 g) and dilute to nearly 00 ml; add $ZnSO_4$ (0·5 g), make up to 100 ml, nd, after some time, filter. Add 10 ml of this eagent to the aq. sample (60 ml) covered with ther (30 ml). In the ether suspend the test paper eshly impregnated with ≃ 0·01 ml of 1% $_4Fe(CN)_6$ soln. followed by ≃ 0·01 ml of 1% nSO_4 soln. Stir for 30 to 60 min. If ≃ 3·6 μg f Fe is present the test spot turns blue; it is un-ffected by 1·8 μg of Fe.

J. P. Stern

1538. **Determination of iron by the impregnation ethod.** A. Dorożalska. *Prace. Kom. Mat.-Przyr. oznań, Tow. Przyj. Nauk*, 1959, **7** (9), 43–49; *Ref. Zhur., Khim.*, 1960, (12), Abstr. No. 47,079.—In the impregnation method, an element is determined by the area of the coloured spot formed when a known volume of the test soln. is placed on paper saturated with an insoluble reagent. This method can be used to determine many elements with an error of ≃ 2% and has many advantages over normal paper chromatography, the error of which is 10 to 30%. The determination of small amounts of Fe^{3+} is described. The impregnated paper is prepared by immersing 8-cm strips of ashless filter-paper in 0·03N-$K_4Fe(CN)_6$ to a depth of ≃ 2 mm. When the soln. reaches the upper edge of the strip, the paper is removed, dried at 60°, steeped in 0·03N-$Zn(NO_3)_2$, washed with water, again dried, and cut into 1·5-cm strips. The test soln. (0·001 to 0·005 ml) is placed on such a strip 1·5 cm from the lower edge, and the lower end of the strip is immersed to a depth of ≃ 2 mm in 0·02N-HCl. The HCl rises up the paper strip, and causes an even diffusion of the Fe^{3+}, thus producing a pale-blue spot, the area of which is proportional to the concn. of Fe. With very dilute soln. the Fe is first concentrated, with simultaneous removal of any other ions present. The method can be used to determine ⩽ 0·1 μg of Fe^{3+} per ml with an error ⩾ 3%.

C. D. Kopkin

1539. **Spectrographic method for the quantitative determination of iron in veined quartz.** L. D. Svirskiĭ and V. L. Zhebunov. *Trudy Khar'kovsk. Politekh. Inst.*, 1959, **31** (1), 147–151; *Ref. Zhur., Khim.*, 1960, (18), Abstr. No. 73,093.—*Procedure*—Place the sample (15 mg), ground in a copper mortar, in a cavity (diam. 3 mm, depth 2·8 mm) in a carbon electrode, and excite the spectra in an arc discharge at 8 amp. with a carbon upper electrode. Evaporate the sample for 7 min. Photograph the spectra in the large spectrograph KS-55. The analysis is carried out by the photometric interpolation method from the lines Fe 3020·6 and Si 3019·97 A. Prepare standards from rock crystal by adding $(NH_4)_2SO_4.FeSO_4.6H_2O$, in the concn. range 0·0005 to 1·0%. The kinetics of the evaporation of Fe is studied. The coeff. of variation of the determination is 9·5%. Grinding the samples in an agate mortar causes contamination with iron.

C. D. Kopkin

1540. **Photometric determination of iron as ferric azide.** K. Kapitańczyk, Z. Kurzawa and Z. Prymiński (Dept. Gen. Chem., Inst. Technol., Poznań, Poland). *Chem. Anal., Warsaw*, 1960, **5** (3), 413–417.—The red ferric azide complex is stable and shows maximum extinction at pH 4·5 to 5·5, provided that sufficient NaN_3 is present (0·5 g per 100 ml). At higher pH a turbidity due to basic ferric azide and $Fe(OH)_3$ develops, and mineral acid completely bleaches the colour at pH 4 or less. Copper in excess of 2 mg per litre interferes by forming an olive-coloured complex, and, owing to their colour, Ni, Co and Cr can be tolerated only up to 50 mg per litre. Salts of Bi, Sn, Ti and Pb hydrolyse at pH 4·5 to 5·5, and tartrates, citrates, phosphates and fluorides decolorise the red complex; Cl^-, NO_3^- and SO_4^{2-} do not interfere. Erratic results are obtained if Fe^{2+} are oxidised with H_2O_2, but conc. HNO_3 is a suitable oxidant. The method can be used over the range 0·02 to 2 mg of Fe^{3+} per 100 ml, and the sensitivity and accuracy are nearly equal to those of the thiocyanate method.

P. Brych

1541. **Determination of metal ions by means of TAA [thioacetamide] and EDTA. III. Determination of iron.** G. C. Krijn, A. S. Koster and G. den Boef (Lab. for Anal. Chem., Univ., Amsterdam, Netherlands). *Anal. Chim. Acta*, 1960, **23** (3), 240–245 (in English).—Iron (0·15 to 5 mg) can be determined in the presence of metals of group 2 and group 3. The group-2 metals are pptd. with thioacetamide in HCl soln. and filtered off. EDTA (disodium salt) is added to the filtrate, which is buffered to pH 11, $CaCl_2$ is added as demasking agent and the pptd. hydroxides of Al, Fe and Mn are filtered off. The Fe in the ppt. can be determined by the spectrophotometric method of Schneider and Janko (*Anal. Abstr.*, 1957, **4**, 1544); Co, Ni and Zn remain in soln. The test soln. containing 0·15 to 5 mg of Fe is made slightly acid with conc. HCl in a centrifuge tube and 4 drops of 6% aq. thioacetamide soln. are added. The tube is heated in a water bath for 15 to 30 min. to coagulate the ppt. and the soln. is filtered through a G4 sintered glass filter into a second centrifuge tube. This tube is heated again in the water bath to destroy the excess of thioacetamide, and cooled. Bromine water is added to oxidise the Fe to Fe^{III} and the excess is removed by heating. The soln. is neutralised with 20% aq. KOH soln. and 1 ml of 0·1M-EDTA (disodium salt) is added. The soln. is cooled and buffered to pH 11 with aq. NH_3 - NH_4Cl soln. and 1 ml of 0·3M-$CaCl_2$ is added to precipitate the Fe (with Al and Mn if present). The ppt. is coagulated by heating at 95°, centrifuged and washed; it is then dissolved in a little conc. HCl and transferred to a 100-ml flask; 8 to 10 ml of 0·1M-EDTA (disodium salt) is added, followed by 3 ml of 25% aq. NH_3 and 3 ml of 30% aq. H_2O_2. The soln. is diluted to vol. with water and the extinction of the Fe^{III} - EDTA -H_2O_2 complex is measured at 525 mμ. The colour is sensitive to light and the flask should be kept in the dark except when actually measuring the extinction. H. M.

1542. **Stylometric determination of sulphur in certain types of steel and welds.** A. S. Dem'yanchuk. *Avtomat. Svarka*, 1959, (12), 82–84; *Ref. Zhur., Khim.*, 1960, (12), Abstr. No. 47,122.—The spectra are excited in a condensed spark discharge from generator IG-2 in a complex circuit (current, 3 amp.; capacity, 0·01 μF; inductance, zero; secondary gap, 3 mm; analytical gap, 2 mm). The electrodes are rods of spectrally pure carbon (10 mm diam.) with hemispherical ends. Sulphur is determined on stylometer ST-7, with sparking for 20 to 30 sec., from the lines S 5453·8 and N 5462·62 A. For convenience of photometry, the N line is positioned in the frame so that the edge of the frame is completely covered by the line Fe 5455·61 A, while the S line is outside the frame. A calibration curve of Δl *vs.* log C is constructed. The lower limit for the determination of S is 0·08%; the mean error is $\pm$ 4·6%. The determination of S in pyrites is carried out from the lines S 5639·9 and N 5666·6 A in the concn. range of 40 to 50% with an analytical gap of 1 mm. The S line is positioned within the frame while the N line remains outside it. The electrodes are rods of electrolytic copper shaped at the working end to a cylinder of diam. 1·8 mm. The standards are chemically analysed samples. A determination takes 3 to 5 min. The error is $\pm$ 3%.
C. D. Kopkin

1543. **Photometric determination of chromium in steel and aluminium alloys.** W. D. Nordlin (Grinnell Corp., Providence, R.I., U.S.A.). *Chemi Analyst*, 1960, **49** (3), 78–79, 88.—For the dete mination of Cr in steel, the sample soln. in H_2SO is treated with ascorbic acid to reduce Fe^{III}, and th Cr is caused to react for 30 min. with EDT (disodium salt) at 100°. The extinction is measure at 538 mμ. The pH must be 0·6 to 0·8. Coba ($>$ 20%), W and Mo do not interfere. For Cr i aluminium alloys, the sample is dissolved in 30% fluoroboric acid, which does not attack Cu. Afte filtration of the residue containing the Cu, ascorbi acid and H_2SO_4 are added and the procedure fo steel is followed. P. D. Parr-Richard

1544. **Macro- and micro-determination of trace of cobalt. V. Analysis of 18-8 steel by activatio with thermal neutrons.** D. Monnier, W. Haerd and J. Vogel (Lab. de Chim. Minérale, Univ. Genève). *Helv. Chim. Acta*, 1960, **43** (3), 675–68 (in French).—The determination is carried out b means of the γ-radiation from ^{60}Co after irradiatio of the sample for 15 hr. in a flux of $\simeq 10^{13}$ neutron per sq. cm per sec. Iron interferes, and is remove by ion exchange. For this purpose the irradiate sample is dissolved in aqua regia and the HNO_3 i removed by repeated evaporation to dryness wit HCl. The residue is then dissolved in 9M-HC (100 ml) and an aliquot (1 ml) is diluted wit 9M-HCl (10 ml) and passed through a column o Dowex 1-X8; Ni and Cr pass through and Co an Fe are retained, the Co being then eluted with 4M HCl. For the γ-spectrometry, three procedure are described. (*i*)—After calibration of the instrument with a known standard of ^{60}Co, the height of the ^{60}Co peak for the sample is measured and substituted in an activation formula. For this procedure the conditions of irradiation must be precisely known. (*ii*)—A known amount of a cobalt salt is irradiated side by side with the sample, and the Co in the sample is determined by calculation from the corresponding peak heights. (*iii*)—The ^{58}Co formed from the Ni or Cr in the sample is used as an internal standard. For this procedure the content of Ni or Cr of the sample must be known. The sensitivity, precision, advantages and disadvantages of each method are discussed.

VII. Determination of micro amounts of cobalt in steel by polarography, spectrophotometry and neutron activation. Comparative study. J. Vogel, D. Monnier and W. Haerdi. *Ibid.*, 1960, **43** (5), 1254–1267 (in French).—The three methods are compared. Each requires the preliminary isolation of the Co. This is carried out by preferential adsorption on a Dowex I-X8 column, followed by elution with 4M-HCl. The soln. (after removal of acid) may be polarographed (*i*) with the addition of a further known amount of Co. The spectrophotometric method (*ii*) is based on measurement of the extinction at 520 mμ of the nitroso-R salt complex. (*iii*) The Co may be made radioactive by neutron bombardment of the sample (5 to 15 hr. at 10^{13} neutrons per sq. cm per sec.). and isolated as described before. The γ-ray spectrum may now be used for the determination of Co (*a*) with an internal standard (*i.e.*, a known quantity of Co is added and irradiated) (this is the recommended procedure); (*b*) with a prior calibration, *i.e.*, an external standard (not recommended); or (*c*) with ^{58}Co from the accompanying Ni as internal standard (useful for alloys of known nickel contents). The

nsitivity and precision decrease in the order (*ii*), (*i*), (*i*); for ensuring freedom from interfering aterials, all methods are much the same; for mplicity, γ-spectrometry is best; for speed (*i*) d (*ii*) are best. R. E. E.
J. L. Prosser

1545. **New redox systems. III. Indirect cerimetric determination of cobalt.** F. Vydra and R. ibil (Chem. Inst., Czech. Acad. Sci., Prague). *alanta*, 1960, **5** (2), 92–96 (in English).—Bivalent balt may be determined by oxidation with Fe^{III} the presence of 1,10-phenanthroline (**I**) (*Ibid.*, 59, **3**, 103; 1960, **5**, 44) followed by the visual potentiometric titration of the ferroin formed th standard $Ce(SO_4)_2$ soln. *Procedure*—Dissolve e sample (0·5 g) in aqua regia. Dilute, filter and ash the residue with dil. HCl. Neutralise an quot of the filtrate with 5% NaOH soln. until a pt. forms, then add sufficient dil. HCl to resolve it. Adjust the pH to between 2 and 4, dd 0·1M-**I** (10 ml) and set aside for 15 min. Add M-H_2SO_4 (50 ml), dilute to 200 ml with water and trate potentiometrically with 0·05M- or 0·1M-$e(SO_4)_2$ with platinum and saturated calomel ectrodes; Fe^{3+} must be added if the sample contains less Fe than Co. Up to 2·9 mg of Co can be termined with an error $\gtrless$ 1% in the presence of i (110 mg), Cd (25 mg), Zn (65 mg), Cu (20 mg), o (30 mg), V (45 mg), Fe (90 mg) and W (50 mg); g, Hg and Th must be absent. W. T. Carter

1546. **Co-precipitation of cobalt with tetrammino-ckel and [tetrammino]copper picrates.** P. A. anichev, I. N. Berezina and A. I. Glotova. *Trudy him. i Khim. Tekhnol.*, 1959, (1), 90–93; *Ref. hur., Khim.*, 1960, (18), Abstr. No. 72,971.—The otope ^{60}Co is used. The soln. (1 ml, containing 00 μg of Ni^{2} or Cu^{2+} and 10 μg of Co^{2+}) is placed a centrifuge tube, and 1·5 ml of a 1% soln. of cric acid (**I**) in 1% aq. NH_3 (for Ni) or 4% aq. H_3 (for Cu) is added. After 15 min. and testing r completion of pptn., the ppt. is centrifuged off, ashed ($\times$ 2) with **I** soln., dissolved in 0·5 to 7 ml of HNO_3 (1:1), and the amount of coptd. Co determined. As the concn. of aq. NH_3 sed to prepare the **I** soln. increases from 0·5 to 5%, the co-pptn. of Co with tetramminonickel icrate increases from 57 to 90% and, with tetramminocopper picrate, from 1 to 6%. At high oncn. of aq. NH_3 practically complete co-pptn. Co is achieved with the nickel salt. At a concn. 0·1%, the Co in the sample does not interfere the determination of Cu. From the study of he influence of the time of contact of the ppt. with he mother liquor, the temp., the amount of Co in he sample and other factors on the co-pptn., it is oncluded that the co-pptn. of Co with these icrates is an adsorption process. C. D. Kopkin

1547. **Determination of cobalt in metals and lloys by amperometric titration with potassium rricyanide.** I. Bozsai (Iron and Metal Works, sepel, Hungary). *Magyar Kém. Lapja*, 1960, 5 (9), 423–425.—The application of the conentional method to steel, tool steel, magnet eel, non-ferrous alloys, metallic nickel, electrical sistance materials (Kanthal), stellite, widia ungsten carbide] and enamels is described; Fe^{3+}, i^{2+}, Cr^{3+}, Al^{3+}, Zn^{2+}, Mg^{2+}, Cu^{2+}, Cl^{-}, SO_4^{2-}, O_4^{3-}, NO_3^{-}, ClO_4^{-}, MoO_4^{2-} and VO_3^{-} do not terfere; Mn interferes, and a correction is necesary. The error is $\simeq \pm$ 0·1 mg of Co.
B. Vaszko

1548. **Separation of radio-cobalt from nickel.** R. Mills and M. J. Vernon (Res. School of Phys. Sci., Australian Nat. Univ., Canberra). *Anal. Chim. Acta*, 1960, **23** (4), 388–390 (in English).—The classical cobaltinitrite separation gives > 99% recovery of 5 mg of Co from 10 g of Ni with a standing time of 45 min. T. R. Andrew

1549. **Precipitation of nickel dimethylglyoximate from homogeneous solution.** E. D. Salesin and L. Gordon (Case Inst. of Technol., Cleveland, Ohio, U.S.A.). *Talanta*, 1960, **5** (2), 81–85 (in English).—Dimethylglyoxime is generated in the presence of Ni by causing diacetyl to react with hydroxylamine at pH 7·5. The soln. is set aside at room temp. for $\lessdot$ 1 hr. and then heated to 80° to 90° for 2 hr. The ppt. consists of large, readily collected needle-like crystals; up to 200 mg of Ni may be handled. In the presence of Cu and Co the separation is superior to that obtained by conventional pptn., but low results for Ni are obtained in the presence of Co and Fe together, unless the standing time at room temp. is increased to 24 hr.
W. T. Carter

1550. **Determination of nickel.** P. Spacu and I. Albescu (Acad. R.P.R., Bucharest). *Acad. R.P.R., Stud. Cercet. Chim.*, 1960, **8** (1), 85–90.—The methods proposed are based on the formation of a complex of Ni with proguanil. *Gravimetric procedure*—A soln. containing 5 to 20 mg of Ni, diluted to 100 ml, is treated with excess of aq. NH_3 to form $[Ni(NH_3)_6]^{2+}$, then a 2% soln. of proguanil hydrochloride is added. Pptn. takes place in the cold at a pH of 9 to 10. After 1 hr. the ppt. is collected in an IG4 filter, then washed with boiling water, and aq. ethanol, and dried at 110°. The error on 0·14 to 1·5 mg is < 5%. *Spectrophotometric procedure*—The pptd. complex is dissolved in warm ethanol and the extinction is determined at 430 mμ. The method was used on amounts ranging from 15 to 200 μg and the error varied from − 3·4 to + 2·6%. Details are given for a procedure in the presence of Al, Zn, Fe and Mg. H. Sher

1551. **Analysis of high-purity nickel by use of the carbonyl process for concentrating traces of impurities.** D. M. Shvarts. *Zavod. Lab.*, 1960, **26** (8), 966–971.—The Ni in a 5-g sample is volatilised as $Ni(CO)_4$ and the residue is analysed spectrographically for Bi, Cd, Sn, Sb, Pb, Zn, As, Mn, Cu and Co. The method is suitable for determining contents of $\simeq 10^{-7}$ to 10^{-5} %. Special apparatus is described. G. S. Smith

1552. **Spectrographic determination of magnesium in cathode nickel.** W. Bykowski and J. Trawiński (Rosa Luxemburg Electric Lamp Works, Warsaw). *Chem. Anal., Warsaw*, 1960, **5** (3), 361–367.—The procedure is based on the method of Nielsch (*Metallkunde*, 1952, **43**, 370); a Fuessner condensed spark is used, with conc. HNO_3 as sample solvent. The carrier electrode is impregnated with 3% polystyrene in benzene. Copper, Fe, Mn and Si (< 0·2%) do not interfere. P. Brych

1553. **Fluorimetric determination of ruthenium.** H. Veening and W. W. Brandt (Purdue Univ., Lafayette, Ind., U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1426–1428.—Bivalent Ru (0·3 to 2 μg) is determined as the complex with 5-methyl-1,10-phenanthroline (**I**) in the presence of Os ($\gtrless$ 25 μg) and of other platinum metals. Results are accurate to within $\pm$ 2%. The insol. complex of Pd with **I** has to be removed by centrifuging; Fe^{III}, Ce^{IV},

Mn^{II}, MnO_4^-, Ag and $Cr_2O_7^{2-}$ interfere. *Procedure*—To the platinum-metals soln., free from strong oxidising agents and from Fe, add 0·1% **I** soln. (15 ml), 20% NaCl soln. (20 ml) and 10% hydroxyammonium chloride soln. (5 ml) and adjust the pH to 6·0 with 5M-NaOH or HCl. Heat the soln. under reflux for 3 hr., then measure the fluorescence intensity in a 1-cm cell at 578 mμ. Determine the Ru content by comparison with a calibration curve. J. P. STERN

1554. **Rapid determination of rhodium.** I. Dema and V. Voicu (Inst. Atom. Phys., Bucharest). *Acad. R.P.R., Stud. Cercet. Chim.*, 1960, **8** (2), 323–328.—The proposed method is based on the production of the complex $[Rh(NO_2)_6][Co(NH_3)_6]$. *Procedure*—The neutral or slightly acid soln. containing 0·01 to 0·1 g of Rh per 100 ml is heated until almost boiling, then treated with excess of solid $NaNO_2$ till the colour changes from red to yellow, followed by dropwise addition of a warm satd. soln. of $[Co(NH_3)_6](NO_3)_3$ until the colour changes to orange. The mixture is cooled; the fine yellow cryst. ppt. is collected in an IG4 crucible, washed with 4 to 5 ml of abs. ethanol and 2 to 4 times with ether, and dried in a vacuum desiccator for 10 to 15 min. The error is $\gtrsim \pm 1·0\%$. Bivalent Pd, Pb^{4+}, Os^{4+}, Fe^{3+}, Ni^{2+} and Cu^{2+} do not interfere; Ir^{3+} and Co^{2+} interfere; Ru must be removed before the determination. H. SHER

1555. **Thiourea complexes of some noble metals. A polarographic determination of rhodium.** F. Pantani and P. G. Desideri (Inst. of Anal Chem., Univ. Florence, Italy). *Talanta*, 1960, **5** (2), 69–74 (in English).—The thiourea complex of Rh^{III} gives a polarographic wave at $E_{\frac{1}{2}} = \simeq -0·40$ V *vs.* the S.C.E. The supporting electrolyte is 0·5M in KCl and 0·2M in thiourea, and the soln. is heated for 10 min. on a boiling-water bath before analysis to stabilise the complex. Gelatin is added as a maximum suppressor and reproducible results are obtained in neutral or slightly acid soln. containing no SO_4^{2-} or NO_3^-. Platinum interferes, but an 8-fold excess of Ir over Rh has no effect; Pd gives a separate wave not due to a thiourea complex, and it is possible to determine Pd and Rh simultaneously with an accuracy of $\simeq 3\%$. W. T. CARTER

1556. **Analytical chemistry of palladium. I. Gravimetric determination of palladium as di-iodo-complexes.** V. Voicu and I. Dema (Inst. Atom. Phys., Bucharest). *Acad. R.P.R., Stud. Cercet. Chim.*, 1960, **8** (2), 301–310.—Three new methods are proposed, based on the production of complexes with KI and pyridine, aniline or quinoline of the form (PdX_2I_2), where X is the base. (*i*) *Procedure with pyridine*—The slightly acid soln. containing 0·01 to 0·1 g of $PdCl_2$ is treated dropwise with pyridine until decolorised, and then with 0·01 to 1 g of KI. The yellow cryst. ppt. is collected after 5 to 10 min. in an IG3 or 4 crucible, and washed with a mixture of 0·05 to 1 g of KI and 2 to 5 drops of pyridine diluted to 100 ml. The ppt. is suction-dried, washed with 3 to 4 ml of abs. ethanol, then 2 to 3 times with ether, and dried for 10 to 12 min. in a vacuum desiccator. The error is $\gtrsim \pm 0·25\%$ on 0·04 g. (*ii*) *Procedure with aniline*—The dil. acid (0·01N to 0·05N-HCl) soln. of 0·01 to 0·1 g of $PdCl_2$ is boiled, then treated with an excess of a 30% ethanolic soln. of aniline until the soln. turns pale yellow, followed by 0·01 to 0·1 g of KI. The soln. is cooled and the yellow-orange ppt. is treated as described above, the wash liquid containing 0·05 to 0·1 g of KI per 100 ml of water. The error is $\gtrsim 0·25\%$ on 0·04 g. (*iii*) *Procedure with quinoline*—The boiling soln. (as in the procedure with aniline) is treated with a 30% ethanolic soln. of quinoline until the soln. is faintly yellow, and then with 0·01 to 0·1 g of KI. The yellow - orange ppt. is treated as described above. The error is $\gtrsim 2·0\%$ on 0·16 g. Iridium and Rh do not interfere, but $[PtCl_6]^{2-}$, Os, Fe, Cu and Co interfere. It is claimed that the method requires only 30 to 40 min.

II. Gravimetric determination of palladium as thiocyanato-complexes. I. Dema and V. Voicu *Ibid.*, 1960, **8** (2), 311–316.—The two methods proposed are based on the production of complexes with pyridine or quinoline of the form $[PdX_2(SCN)_2]$. *Procedure with pyridine*—Pyridine is added until the soln. (50 to 70 ml, containing 0·01 to 0·05 g of Pd) is decolorised, followed by 0·1 g of NH_4SCN. The yellow pearly ppt. settles in 5 to 10 min., is collected in an IG4 filter and washed with a soln. containing 2 to 3 drops of pyridine and 0·06 to 0·1 g of NH_4SCN in 100 ml of water. The ppt. is suction-dried, washed with 1 to 2 ml of abs. ethanol and once or twice with ether, and vacuum-dried. *Procedure with quinoline*—To the sample soln. heated to 50° to 60° are added 15 to 20 ml of 50% ethanol, a 50% ethanolic soln. of quinoline until the soln. turns slightly yellow, and 0·1 g of NH_4SCN. The mixture is stirred until a yellow - orange cryst. ppt. settles. This is cooled, and collected as described above, but washed with a 0·08% aq. soln. of NH_4SCN, then suction-dried, and washed with 3 to 5 ml of abs. ethanol and 2 to 3 times with ether and vacuum-dried. The error in each procedure is $\gtrsim 1·0\%$.

III. Gravimetric determination of palladium with pyridine and potassium dichromate. V. Voicu and I. Dema. *Ibid.*, 1960, **8** (2), 317–321.—The proposed method is based on the production of $[PdPy_4][Cr_2O_7]$. In the absence of Rh^{3+}, Ni^{2+}, Co^{2+} and Mn^{2+}, 50 ml of the soln. containing 0·005 to 0·05 g of Pd at pH 4 to 6·5 is boiled and treated with pyridine until the yellow - orange colour is discharged, and then with an excess of a satd. soln. of $K_2Cr_2O_7$ until the mixture is yellow. After cooling the soln., the yellow cryst. ppt. is collected in an IG3 or 4 crucible, and washed first with 2 to 4 ml of a satd. soln. of $K_2Cr_2O_7$ plus 0·5 ml of pyridine in 100 ml of water, then 3 to 5 times with 1 ml of abs. ethanol and 2 to 3 times with ether, and dried in a vacuum desiccator for 10 to 15 min. The error is $\gtrsim \pm 1·5\%$. In the presence of Rh^{3+}, Ni^{2+}, Co^{2+} or Mn^{2+}, the soln. treated with pyridine is further treated with $\simeq 0·1$ g of EDTA and heated on a water bath for 10 to 15 min., after which $K_2Cr_2O_7$ is added as described above. The error is $\gtrsim \pm 2\%$. Bivalent Cu, Cd^{2+} and $[PtCl_6]^{2-}$ interfere. H. SHER

1557. **Colorimetric determination of palladium in uranium fission element alloys.** L. E. Ross, G. Kesser and E. T. Kucera (Chem. Engng Div., Argonne Nat. Lab., Ill., U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1367–1368.—*Procedure*—To the sample soln. (30 ml, containing 5 to 25 μg of Pd) in a 60-ml separating-funnel add EDTA (disodium salt) soln. (3%) (2 ml), adjust the pH to 1·0 to 2·0, add 1-nitroso-2-naphthol soln. (1%) (0·1 ml), set aside for 10 min. and extract with 10 ml of toluene. Wash the toluene extract with N-NaOH (10 ml) and centrifuge the organic layer to remove droplets. Measure the extinction of the toluene layer at 370 mμ. T. R. ANDREW

1558. **Photometric determination of palladium in the products of nickel production.** S. E. Kreĭmer, L. P. Butylkin and A. V. Stogova ("Severonickel" Combine, U.S.S.R.). *Zhur. Anal. Khim.*, 1960, **15** (4), 467–471.—It is shown that an exchange reaction takes place between soln. of palladium dimethylglyoximate (**I**) in $CHCl_3$ and aq. diantipyrinylmethane (**II**) and aq. KI soln. with the probable formation of the complex $(C_{23}H_{24}O_2N_4)_2.H_2[PdI_4]$. Add aqua regia (30 ml) to nickel tailings (0·5 g), heat the mixture and evaporate almost to dryness; treat the residue twice with conc. HCl and again evaporate. Add H_2O to the residue, filter off the insol. portion, wash it with hot H_2O, and ignite it with the filter-paper; fuse the residue with $K_2S_2O_7$ and extract the melt with H_2O. Add this soln. to the main soln. and reduce the vol. to 100 ml by evaporation. Add conc. HCl (3 ml) and 1% ethanolic dimethylglyoxime soln. (10 ml), stir vigorously and set the mixture aside for 15 min. Extract with $CHCl_3$ (10 and 5 ml), combine the extracts and wash them with 0·2N-HCl (2 or 3 × 25 ml). Add 40% KI soln. (10 ml) and 2% **II** soln. (20 ml) in 0·5N-HCl and shake for 2 min. Run off the $CHCl_3$ layer, add $CHCl_3$ (2 ml) to the remaining soln., shake vigorously and run off the $CHCl_3$ layer. Combine the extracts, wash them as before and dilute them to 25 ml with $CHCl_3$. The cherry-red colour is measured against a standard at 450 mμ. For an alternative photometric determination of Pd, evaporate the $CHCl_3$ soln. of **I** to dryness on a water bath, decompose **I** by heating with aqua regia (25 ml), evaporate to dryness, add conc. HCl (10 ml) and evaporate almost to dryness. Dissolve the chlorides by heating with H_2O, add 0·1% nitroso-R-salt soln. (10 ml), heat to boiling, cool, and dilute the soln. to 100 ml. Measure the extinction with a green filter. This method is suitable for the determination of Pd in tailings from nickel production containing ⩾ 45% of Ni, ⩾ 20% of Cu, and ⩾ 5% of Fe. The method is modified for products containing a comparatively large amount of Fe. K. R. Cook

1559. **Spectrophotometric determination of palladium with *o*-mercaptobenzoic acid.** I. Dema and V. Voicu (Inst. Atom. Phys., Bucharest). *Acad. R.P.R., Stud. Cercet. Chim.*, 1960, **8** (1), 173–178.—A soln. of the sample in 50% acetone at a pH between 2 and 4 containing < 2·6 μg of Pd per ml is treated with 5 drops of an ethanolic soln. of *o*-mercaptobenzoic acid. The max. extinction is at 428 mμ. There is no interference from Fe^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Pb^{2+}, Sn^{2+}, Cd^{2+}, Al^{3+}, Au^{3+}, Rh^{3+} (< 1:2), TeO_4^{3-} or Au^{+}, but Pt^{4+}, Pt^{2+}, Rh^{3+} (> 1:2), Cr^{3+} (in large amounts), I^- and oxidising agents interfere. H. Sher

1560. **Separation of palladium from niobium and zirconium.** J. F. Duke and W. Stawpert (Nat. Phys. Lab., Teddington, Middx., England). *Analyst*, 1960, **85**, 671–672.—The method is based on the ability of Pd to form a complex iodide, PdI_4^-, whereas neither Nb nor Zr forms a stable iodide in aq. soln., and the extraction of this iodide into isobutyl methyl ketone. Alloys of Pd and Zr are dissolved in aqua regia and fumed with H_2SO_4. Alloys of Pd and Nb are fused with $KHSO_4$ and the residue is dissolved in oxalic acid. Sulphuric acid and KI are then added, the KI to an excess of 10% over that required to precipitate the palladous iodide. A vol. of isobutyl methyl ketone equal to that of the aq. phase is added and the mixture is shaken intermittently until the colour of the aq. layer is discharged. The separated organic layer is back-washed with a little H_2SO_4 (20%), and the separation of Pd from Nb and Zr is then complete. After removal of solvent and destruction of organic matter with HNO_3, the Pd is determined gravimetrically with dimethylglyoxime. The Nb and Zr in the aq. layer are determined by pptn. with cupferron and subsequent ignition to the oxides. A. O. Jones

1561. **Organic reagents for osmium.** G. Baiulescu, C. Lazar and C. Cristescu (Parhon Univ., Bucharest). *Zhur. Anal. Khim.*, 1960, **15** (4), 505–506.—4-Acetamidobenzaldehyde thiosemicarbazone (**I**) is used as a 0·1% soln. in 1% NaOH soln. and 4-nitrobenzaldehyde thiosemicarbazone (**II**) is used as a 0·05% soln. in 1% NaOH soln. An H_2SO_4 soln. (conc. H_2SO_4 - H_2O) (8:17) (1 ml) is added to 0·1 to 3·0 ml of the osmium soln., then **I** or **II** soln. (2 ml) is added and the mixture is set aside, for 5 min. with **I** and for 15 min. with **II**; each soln. is then diluted to 10 ml with ethanol. A yellow - brown colour is obtained with **I** and a red - brown colour with **II**. The max. extinction in both cases is measured at 436 mμ in a Pulfrich photometer. The use of **I** and **II** enables 1·2 to 14·7 μg of Os per ml and 0·49 to 8·60 μg of Os per ml, respectively, to be determined. These reagents are more sensitive than thiourea. K. R. Cook

1562. **Neutron activation analysis for iridium in platinum.** L. S. Jowanovitz, F. B. McNatt, R. E. McCarley and D. S. Martin, jun. (Inst. for Atomic Res., Iowa State Univ., Ames, U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1270–1272.—The sample, after irradiation, is dissolved in aqua regia and Ir is then pptd. as the hydroxide and counted with a scintillation spectrometer. Hydrated IrO_2 is used as an external standard; the use of ^{195m}Pt as an internal standard is not recommended. The coeff. of variation reported for a 0·04-p.p.m. sample (7 replicates) is 10%. K. A. Proctor

1563. **Spectrographic determination of impurities in iridium and rhodium.** A. A. Kuranov, V. D. Ponomareva and N. I. Chentsova. *Zhur. Anal. Khim.*, 1960, **15** (4), 476–480.—It is shown that reference standards for spectrographic analysis of iridium and rhodium can be prepared from metal powders. A method is described for the determination of Pt, Au, Pd, Ru, Fe, Ni, Al, Cu, Si, Pb, Ba and Rh in iridium and the first 11 elements and Ir in rhodium. Graphite powder (0·05 g) is added to each of three 0·1-g portions of metal powder (iridium or rhodium), each mixture is ground carefully and then compressed in a steel matrix (diam. 4 mm) under pressure (4000 to 5000 kg per sq. cm). A spectrograph with a triple-lens system, a slit width of 0·017 mm for the analysis of iridium and 0·025 mm for rhodium, a lower rod-shaped graphite electrode (diam. 6 mm) with a truncated conical tip, an electrode gap of 2·5 mm and an a.c. arc with an 8-amp. current as an excitation - activation source, are used. The exposure time is 60 sec. All standards and samples are photographed three times through a three-stage attenuator. Type-I spectrographic plates are used for the long-wave region and type-II plates for the short-wave region. K. R. Cook

1564. **Determination of free acid in the presence of hydrolysable cations.** A. Moscowitz, J. Dasher and H. W. Jamison, jun. (Crucible Steel Co. of America, Pittsburgh, Pa.). *Anal. Chem.*, 1960, **32**

(10), 1362–1364.—A simple method is given for the determination of free acid in the liquors resulting from the pickling of stainless-, silicon-, alloy- and carbon-steels and of titanium with HNO_3, H_2SO_4 and HNO_3 - HF, and in similar soln. In favourable cases, the error is $< \pm 2\%$. *Procedure*—Dilute to 50 ml an aliquot containing 1 to 10 milli-equiv. of free acid. If the sample is an iron-pickling liquor, add 11·6% $Fe(NO_3)_3.9H_2O$ soln. (0·2 ml). If the sample does not contain HF, add also 32% NH_4F soln. (0·6 ml). If the sample is a titanium-pickling liquor, add $Fe(NO_3)_3$ soln. (2 ml) and NH_4F soln. (0·6 ml). Titrate with 0·2N-NaOH to the first visible red - brown ppt.; the soln. turns yellowish just before the end-point.

A. R. ROGERS

1565. **Analysis of small samples of monazites with a gamma scintillation spectrometer.** G. H. M. Couwenberg, C. L. D. Kooy and A. E. Korvezee (Lab. of Phys. Chem., Tech. Univ., Delft, Holland). *Rec. Trav. Chim. Pays-Bas*, 1960, **79** (8), 895–909 (in English).—The γ-ray spectra of a number of specimens of known radioactive-element content have been examined and the information is used to determine the U and Th contents of monazites. The results obtained for low U contents are not as good as with chemical analysis. In leached ores, where the radioactive equilibrium with Ra, etc., has been disturbed, the results are only approximate. E. J. H. BIRCH

1566. **Determination of arsenic in coal and coke.** S. R. Crook and S. Wald (Nat. Coal Board, Scientific Dept., Albert Embankment, London). *Fuel, London*, 1960, **39** (4), 313–322.—Methods for the determination of As in anthracite have been examined in several laboratories during the revision of BS 1016. In the method adopted, the ground sample is mixed with MgO and $KMnO_4$ and burned in O. The As is extracted with acid and reduced to AsH_3, which is then absorbed and oxidised by means of iodine soln. The intensity of the molybdenum blue colour developed by the addition of ammonium molybdate and hydrazine sulphate is measured and the As is determined by reference to a calibration curve. Repeatability and reproducibility are both 1·0 p.p.m. of As_2O_3 for samples containing < 6 p.p.m., and 1·5 p.p.m. for samples with > 6 p.p.m. The method is also applicable to coal and coke and can be used for the simultaneous determination of P. The paper-stain finish, although less precise, is also to be retained in BS 1016. G. BURGER

1567. **Free cyanide analysis of copper cyanide plating solutions.** J. R. Crain (Westinghouse Electric Corp., East Pittsburgh, Pa., U.S.A.). *Plating*, 1960, **47** (9), 1032–1035.—For the most accurate value of "free" cyanide, the cold method is recommended; the ice must be weighed out accurately since $[CN]^-$ is affected by changes in dilution. Whilst the rate at which the "free" cyanide content is increased, by dissociation of $[Cu(CN)_3]^{2-}$, is negligible at the temp. of the cold method (3° to 5°), this effect is increasingly important at higher temp. and causes high results. The end-point is more difficult to detect at higher temp. and the increased titration time could also introduce errors. J. H. WATON

See also Abstracts—**1341**, Determination of gases in metals. **1346**, Indicator for mercurimetry of halides. **1350**, Pptn. of Cu - Sn group with H_2S. **1351**, **1352**, Ion exchange for qual. separation of metals. **1661**, Determination of K in presence of NH_4^+. **1642**, Fe, Ni and V in petroleum. **1663**, Metallochromic indicator for Ca. **1733**, **1734**, Be in air. **1739**, Be in natural water. **1775**, Apparatus for determination of hydrazine in soln. **1776**, O in titanium and steel.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather and explosives.

1568. **Preliminary examination of organic compounds yielding water and ammonia by spot-test analysis.** F. Feigl and J. R. Amaral (Lab. da Prod. Mineral, Min. da Agric., Rio de Janeiro, Brazil). *Mikrochim. Acta*, 1960, (5–6), 816–820 (in German).—The detection of 8 classes of compound that yield NH_3 or H_2O on pyrolysis is described. By heating these at 120° to 180° with thiobarbituric acid (**I**) or Michler's thioketone, H_2S is liberated. *Procedure*—Add a little solid **I** and 1 to 2 drops of ethanol or benzene to a drop of test soln. in a micro test-tube. Evaporate to dryness and place the tube with a piece of Pb acetate paper over the open end in a glycerol bath at 110°. Heat gradually to 180°; a positive reaction is indicated by the appearance of a black or brown stain in a few minutes. The limit of detectability is 0·3 to 50 μg. A test with hexamine and Nessler reagent for compounds yielding water only is also described. P. D. PARR-RICHARD

1569. **Infra-red analysis, alone and combined with other techniques, as a tool in the determination of the structure of organic compounds.** C. la Lau (Koninkl. Shell Lab., Amsterdam, Netherlands). *Chem. Weekbl.*, 1960, **56** (39), 537–541.—Examples are given of the use of i.r. spectrophotometry, alone and in combination with u.v. spectrophotometry, Raman spectroscopy and mass spectrometry.

M. J. MAURICE

1570. **The formation of active complexes in the oxidation layer during elementary analysis.** G. Kainz and H. Horwatitsch (Anal. Inst. der Univ. Wien, Austria). *Mikrochim. Acta*, 1960, (5–6), 917–926 (in German).—Several compounds were submitted to combustion over CuO at different temp. under N and the amount of CO_2 formed was measured. Ease of oxidation was in the order CO, acetic acid, acetylene, petroleum, ethylene, benzoic acid, xylene, toluene, benzene and methane. Except for homologues, the b.p. was no criterion of ease of oxidation. With petroleum, a step-wise reaction occurred, because of the intermediate formation of methane; the acids behaved similarly, though CO_2 was rapidly formed from the carboxyl group. Combustion of acetylene and ethylene was incomplete. Oxidation occurs by the formation of cleavage products which are successively adsorbed and desorbed by CuO until oxidation is complete; methane has the lowest retention time and thus resists oxidation most strongly. The influence of various factors on oxidative efficiency is discussed.

P. D. PARR-RICHARD

571. **Conductimetric micro-determination of bon and hydrogen in organic compounds.** N. E. 'man and Ven-Yun Van (Inst. of Organic ments and Compounds, Acad. Sci., Moscow). *ır. Anal. Khim.*, 1960, **15** (4), 487–494.—After olytic combustion of the organic sample in a id stream of O, the H_2O obtained is frozen with nixture of solid CO_2 and acetone, and the CO_2 ained is passed into an electroconductivity cell which it is absorbed by dil. NaOH or $Ca(OH)_2$ ı. The frozen H_2O is evaporated in a stream of or N on to a layer of hot platinised carbon black, CO formed is then oxidised over heated CuO, l the resulting CO_2 is passed into an electro- ductivity cell containing a dil. soln. of alkali. e C and H are measured by the change in ductivity of the absorbing soln. The repro- ibility of results is more satisfactory than with gravimetric method and, except for weighing sample, the entire procedure can be made fully omatic. It can be used for the determination solids and liquids containing N, halogens (in- ding F), S, B, Si, P and metals. Recoveries for umber of organic substances are tabulated.
K. R. Cook

572. **Simultaneous micro-determination of bon, hydrogen and halide.** A. S. Zabrodina and F. Egorova. *Vestn. Moskov. Univ., Ser. Khim.*, 60, II, No. 4, 66–70.—Metallic copper at a nperature of 10° to 15° quant. absorbs Br and Cl. Cl-containing compound, when burnt in a stream O, produces a significant amount of HCl; CuO therefore mixed with the metallic copper to ct with the HCl to form H_2O, which is absorbed anhydrone before and after the combustion ducts are passed over the copper - CuO mixture. drogen bromide is not produced in significant ounts when Br-containing compounds are burnt. e accuracy of the determination for C and H is hin ± 0·2% and for halides within ± 0·4%.
A. Burwood-Smith

573. **Sodium dispersions in elementary analysis.** Patrick and F. Schneider (C.W. Post College, ookville, N.Y., U.S.A.). *Mikrochim. Acta*, 1960, 6), 970–972 (in English).—The conventional ion method for detecting N, halogens, S, etc., organic compounds is modified by using a persion of sodium in toluene; this serves not y to decompose the sample but to reduce any to NH_3. *Procedure*—Place 1 μg to 1 mg of stance into a capillary tube sealed at one end l add 5 to 10 times as much of the dispersion. ce a piece of red litmus paper over the open end l warm the tube. To identify S, destroy the ess of sodium with ethanol, transfer the soln. to lean tube and acidify with HNO_3. Detect H_2S h Pb acetate paper. P. D. Parr-Richard

574. **Automatic chloride analyser.** D. M. ılson and L. A. Cavanagh (Stanford Res. Inst., nlo Park, Calif., U.S.A.). *Anal. Chem.*, 1960, (10), 1245–1247.—A completely automatic thod for the coulometric titration of I^-, Br^- and in combustible samples is described. Small nples (0·5 to 1 mg) can be analysed in 10 min. h an accuracy greater than that by other thods. K. A. Proctor

575. **Micro-determination of nitrogen based on Dumas method. Improvements in the Shelberg l Schöniger technique.** R. Lévy and B. Cousin ntre Nat. de la Rech. Sci., 39 bis rue de Dantzig, Paris). *Mikrochim. Acta*, 1960, (5–6), 854–862 (in French).—Modifications to Schöniger's apparatus (*Mikrochem.*, 1952, **39**, 229) are described and illustrated. These consist of a simplified gas-flow path, a readily replaceable packing, more flexible connections and movable electric furnaces which can be opened. The method of use, described in detail, gave good precision with, for example, tryptophan, and certain systematic errors have been eliminated. P. D. Parr-Richard

1576. **Ultra-micro determination of nitrogen in samples of organic substances weighing not more than 0·5 mg.** G. Gutbier and M. Boëtius (Tech. Hochschule, Dresden, Germany). *Mikrochim. Acta*, 1960, (5–6), 636–640 (in German).—The determination of N in 0·1 to 0·5-mg samples is possible by the Dumas method with a miniature Pregl apparatus. A CO_2 regulator is incorporated to permit reproducible blanks to be obtained. Errors are from + 0·3 to − 0·1%. J. P. Stern

1577. **Determination of small amounts of nitrogen in organic material. Elimination of traces of nitrogen from reagents and protection therefrom during the analysis.** H. Roth (Agric. Exp. Sta., B.A.S.F., Limburgerhof, Germany). *Mikrochim. Acta*, 1960, (5–6), 663–669 (in German).—The N (≃ 10 p.p.m.) is determined by the Kjeldahl technique and measured photometrically by means of Nessler reagent in an ELKO II photometer with a S42 filter. For such small amounts of N, large samples (1 to 2 g) are required; also, blanks must be kept low and constant both by purification (described) of reagents and by excluding atmospheric basic N compounds by means of a water-filled U-tube. By this means blanks equiv. to 4 to 6 μg of N are reproducibly attained with 20 ml of soln. The need for repeated blank determinations is thus eliminated; alternatively, the photometric reading is taken against a blank determination and the blank value thus eliminated.
J. P. Stern

1578. **Rapid volumetric micro-determination of phosphorus in organic substances.** R. Püschel and H. Wittmann (Univ., Graz, Austria). *Mikrochim. Acta*, 1960, (5–6), 670–674 (in German).—The Schöniger combustion method is followed by direct cerimetry (with 0·005M-Ce^{3+}) of the PO_4^{3-} produced, with Eriochrome black T (**I**) as indicator. Errors are < 0·3% of P and maximum deviations of duplicate determinations on 3-mg samples are within ± 0·2%; F, As, Si or metals forming complexes with **I** interfere. J. P. Stern

1579. **Application of the flask combustion method to the micro-determination of phosphorus in organic substances.** A. Dirscherl and F. Erne (F. Hoffmann-La Roche and Co. A.-G., Basel, Switzerland). *Mikrochim. Acta*, 1960, (5–6), 775–788 (in German). —Loss of P in the Schöniger combustion procedure is ascribed to its retention in carbon deposited on the platinum gauze, and is avoided by the use of a larger flask and by the addition of $(NH_4)_2S_2O_8$ to the sample. J. P. Stern

1580. **Simultaneous determination of traces of selenium and mercury in organic compounds by X-ray fluorescence.** E. C. Olson and J. W. Shell (The Upjohn Co., Kalamazoo, Mich., U.S.A.). *Anal. Chim. Acta*, 1960, **23** (3), 219–224 (in English). —Solid samples (600 mg) are weighed into a special aluminium plate, having a depression in which the

sample is compressed into a block with the aid of a hydraulic press. Liquid samples are irradiated in an aluminium cell with a Mylar window. Irradiation is with a tungsten-target X-ray tube operated at 50 kV and 45 mA. A lithium fluoride analysing crystal and a Tl-activated sodium iodide scintillation counter are used. The intensity of the L_α line at 36·00° 2θ is used for the determination of Hg and the K_α line at 31·88° 2θ for Se. The intensity of the L_β line of W is also measured, and the ratios of the intensities of the Se and Hg lines, respectively, to the W line are calculated. The values found are compared with a calibration graph prepared from standard samples treated in a similar manner. The intensities are found by measuring the time required for 10^6 counts. The method is claimed to give results accurate to $\pm$ 1 p.p.m. for samples containing 2 to 40 p.p.m. of Se or Hg.

H. M.

1581. **Qualitative functional-group analysis of gas-chromatographic effluents.** J. T. Walsh and C. Merritt, jun. (Pioneering Res. Div., Quartermaster Res. and Engng Centre, U.S. Army, Natick, Mass.). *Anal. Chem.*, 1960, **32** (11), 1378–1381.—A method of qual. gas-chromatographic analysis is described in which retention-time data are combined with functional-group colorimetric tests. The method is rapid and simple, and requires little extra time and no expensive supplementary instrumentation. The technique can also be used to decide whether a chromatographic peak consists of one or more components. C. B. Baines

1582. **Acetyl-group determination using acetic anhydride labelled with carbon-14.** R. H. Benson and R. B. Turner (Radioisotope Res. Lab., Veterans Admin. Hospital, Houston, Texas, U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1464–1465.—The sample is acetylated in pyridine by heating with labelled acetic anhydride (prep. described) (2 millimoles per expected hydroxyl group for 1 millimole of sample) at 125° for 4 hr. The product is poured into H_2O and ethyl ether is added. The ether layer is extracted successively with dil. HCl, dil. NaOH soln., H_2O and satd. NaCl soln. After evaporation of the ether, the product is purified by chromatography and recrystallisation. Alternatively, the acetylation can be carried out in the presence of labelled Na acetate as a catalyst instead of in pyridine. A weighed amount of the derivative is dissolved in dimethylformamide, and the specific activity is determined in a flow counter. The number of acetyl groups is obtained by dividing by two the value obtained for the specific activity of the acetic anhydride. G. Burger

1583. **Infra-red absorption spectrum of methane from 2470 to 3200 cm^{-1}.** E. K. Plyler, E. D. Tidwell and L. R. Blaine (Nat. Bur. Stand., Washington, D.C.). *J. Res. Nat. Bur. Stand., A*, 1960, **64** (3), 191–212.—A total of 2460 lines were measured in this region. The observed spectrum is shown in five charts.

1584. **Gas - solid chromatographic separation of some light hydrocarbons.** T. A. McKenna, jun., and J. A. Idleman (Firestone Synthetic Rubber and Latex Co., Orange, Tex., U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1299–1301.—Separate the saturated and ethylenic hydrocarbons up to C_4 by passage successively through a 10-ft. column of 60 to 80-mesh silica (Davison Grade 62, previously heated at 310° for 1 hr.) and a 10-ft. column of 60 to 80-mesh alumina (Alcoa Grade F-10, previous heated overnight at 110°), at a temp. in the ran 30° to 90°, with He as carrier gas at 150 ml p min. Before use, the columns must be purged wi He until all water is eluted (up to 24 hr.). Altern tively, use a 30-ft. column, containing 20·8% propylene carbonate on alumina, at room temp.

A. R. Rogers

1585. **Hydrogenation as an aid in the identific tion of unsaturated hydrocarbons by gas chrom tography.** B. Smith and R. Ohlson (Inst. Organisk Kemi, Chalmers Tekn. Högskola, Göt borg, Sweden). *Acta Chem. Scand.*, 1960, **14** (6 1317–1324.—Methods are described for the comple hydrogenation of unsaturated hydrocarbons a the stepwise hydrogenation of alkadienes and alkynes in fractions from gas-chromatograph analyses. These are particularly useful in t identification of dienes and alkynes, and also perm the detection of unsaturated hydrocarbons specific chromatographic bands and the conversi of unseparable unsaturated hydrocarbon mixtur into separable saturated mixtures, *e.g.*, mixtur of 3- and 4-methylpentene were hydrogenated 3- and 2-methylpentane and subsequently separat on squalane. S. M. Marsh

1586. **Determination of chloroform in aqueo solution.** E. A. Robinson (A. R. Davis & Co. Lt Croydon, Surrey, England). *Anal. Chim. Ac* 1960, **23** (4), 305–308 (in English).—The hydroly of $CHCl_3$ by aq. soln. of alkali has been studied a function of alkali concn. for NaOH and KOH.

T. R. Andrew

1587. **Rapid determination of 3-chloropropene methoxymercuration.** K. L. Mallik (Coates Che Lab., Louisiana State Univ., Baton Rouge, U.S.A *Anal. Chem.*, 1960, **32** (10), 1369–1370.—To 0·05 or 0·1M-3-chloropropene in methanol add an exce of 0·1M-mercuric acetate in methanol, 0·005M 0·01M in $HClO_4$, set aside at room temp. for min. and titrate with 0·1N-HCl in butanol, wi diphenylcarbazone as indicator. Perform a bla determination. The recoveries in four replica assays ranged from 99·1 to 100·5%.

A. R. Rogers

1588. **Quantitative determination of ethyle epoxide [epoxyethane], propylene epoxide [1,2-epox propane] and higher-molecular-weight epoxid using dodecanethiol.** B. J. Gudzinowicz (Petroche Dept., Nat. Res. Corp., Cambridge, Mass., U.S.A *Anal. Chem.*, 1960, **32** (11), 1520–1522.—A pr cedure is described for the determination of epoxid in mixtures to which other methods cannot applied. The sample, in aq. or organic soln., shaken with a standard soln. of dodecanethiol ethanol and an amount of ethanolic KOH sol slightly in excess of that required to neutralise t sample (separately determined). After 20 mi acetic acid and isopropyl alcohol are added, a the excess of the thiol is titrated with iodine sol The values found are more accurate than tho obtained with the $MgCl_2$ - HCl procedure, but le so than those given by the $HClO_4$ - $NaIO_4$ meth (*cf.* Eastham and Latremouille, *Canad. J. Res.*, 1950, **28**, 264). High concn. of aldehydes interfe

G. Burger

1589. **Determination of C3- and C4-alcohols benzene.** D. E. Nicholson (Humble Oil & Refini Co., Baytown, Tex.). *Anal. Chem.*, 1960, **32** (1

372.—Infra-red data are given for n-propanol, butyl alcohol, isopropyl alcohol and t-butyl cohol in benzene.

1590. **Gas - liquid chromatography of allylic cohols and related branched-chain acids.** G. opják and R. H. Cornforth (Hammersmith Hospi-l, Ducane Road, London, W.12). *J. Chroma-graphy*, 1960, **4** (3), 214–221.—The relative reten-on volumes (R_v) of polyprenols (compounds ontaining isoprenoid units) and related branched-ain acids are reported. Two columns were used, ne containing ethanediol - adipic acid polyester n Celite and one containing Apiezon L on Celite. ll determinations were made at 197° with a 120 cm 4 mm column and a gas-density balance. Identi-cation is reasonably certain when values of R_v for oth columns are used. The separation factors atios of R_v) for compounds differing by specific roups or configurations (*e.g.*, isoprenoid unit or *ans - cis*) are tabulated. R. M. S. Hall

1591. **The catalytic spot-test detection of ether.** . Feigl, R. J. Amaral and D. Haguenauer-Castro Lab. da Prod. Mineral, Min. Agric., Rio de Janeiro, razil). *Mikrochim. Acta*, 1960, (5–6), 821–826 n German).—By rapid evaporation of pure ethyl ther in air and heating at 200°, ether peroxide is ormed, which catalyses the oxidation of Cu acetate - enzidine reagent. Other solvents, *e.g.*, benzene, oluene and CS_2, prevent this reaction, but such nterference is masked by the addition of a small mount of $CHCl_3$ or CCl_4. Certain other aliphatic thers give a positive reaction, but cyclic ethers do ot. *Procedure*—Place a drop of ether in a micro-est-tube and cover the open end with a paper mpregnated with a 1:1 mixture of aq. saturated oln. of Cu acetate and benzidine chloride. Immerse a glycerol bath at 200°; a blue colour appears fter about 1 min. P. D. Parr-Richard

1592. **Determination of chloride ion in ethanediol y pCl measurement.** R. B. LeBlanc and R. T. IcFadden (Texas Div., Dow Chem. Co., Freeport, .S.A.). *Talanta*, 1960, **5** (2), 78–80 (in English).—p to 1 p.p.m. of Cl^- in ethanediol can be deter-ined with silver - silver chloride and satd. calomel lectrodes separated by a KNO_3 salt bridge. The onductivity of the sample (100 ml) is increased by ne addition of 3M-KNO_3 (5 drops) and the cell otential is measured with a pH meter that has reviously been standardised with samples of known l^- content. Propionaldehyde (30 p.p.m.), acetic cid (10 p.p.m.) and Fe (0·5 p.p.m.) are without ffect, but 1 p.p.m. of Fe gives an error of − 0·1 .p.m. of Cl^-. The sensitivity is 0·1 p.p.m. and ne accuracy ± 0·1 p.p.m. W. T. Carter

1593. **Evaluation of crude glycerin.** L. V. Cocks Unilever Ltd., Res. Dept., Port Sunlight, Ches., ngland). *Analyst*, 1960, **85**, 686–687.—The ethod of evaluating glycerin by means of the organic residue," which is the total residue at 60° less the ash, is a tedious analytical process nd the result gives no indication of the presence f impurities volatile at 160°. The value of the o-called "matter-(organic)-non-glycerol" (MONG) gure, which is 100 less the sum of the glycerol by the KIO_4 method), ash and water (by the Karl ischer method), has been examined and 418 sets f figures have been collected to compare MONG ith "organic residue." In quoted examples where MONG is higher than the "organic residue" the difference is shown to be due to the presence of propane-1,3-diol. It is concluded that, in normal crude glycerins, MONG is a figure of the same order as the "organic residue," while in abnormal crude glycerins MONG includes organic impurities (volatile at 160°) that would not be included in the "organic residue" figure, and it is proposed that MONG should be considered as an improved alternative to "organic residue." A. O. Jones

1594. **Colour reactions given by sugars and diphenylamine - aniline spray reagents on paper chromatograms.** R. W. Bailey and E. J. Bourne (Royal Holloway Coll., Englefield Green, Surrey, England). *J. Chromatography*, 1960, **4** (3), 206–213.—The use of the spray reagent of Buchan and Savage (*Analyst*, 1952, **77**, 401) has been extended. Specific colours are given with uronic acids, aldoheptoses and methylpentoses when heated at 80° (5 min.). Specific reactions with pentoses at 60° are not affected by the first two groups of compounds. When the diphenylamine and aniline are successively sprayed, dried and heated instead of being applied as a mixture, specific colour reactions are given with all aldohexoses and reducing aldohexose disaccharides not containing a 1,4 link and also with some ketoses. D-Fructose (red - brown) and L-sorbose (yellow) are clearly differentiated, while furfuraldehyde and 5-hydroxymethylfurfural are not. The substitution of triphenylamine for diphenylamine did not yield useful results. R. M. S. Hall

1595. **Sugar determination according to Luff - Schoorl.** E. L. Krugers Dagneaux and P. Raymond (Lab. N.V.P. de Gruyter en Zn., 's-Hertogenbosch, Netherlands). *Chem. Weekbl.*, 1960, **56** (38), 533–534.—A modification is proposed of the Luff - Schoorl procedure (*Ibid.*, 1929, **26**, 132). The reaction mixture is now kept at 120° for exactly 10 min. A new table is given for lactose. For other sugars the Luff - Schoorl tables can be used. M. J. Maurice

1596. **Qualitative micro-determination of carbonyl compounds by radial chromatography of their hydrazones.** L. Peyron (Antoine Chiris, Grasse, France). *France et ses Parfums*, 1959, **2** (12), 41–48.—Functional carbonyl groups in plant extracts were blocked as acylhydrazones, transformed into coloured arylhydrazones, and identified by radial chromatography. One part of the plant material was mixed with 10 parts of cold carbonyl-free dichloromethane for 10 min. The filtrate from this was heated under reflux for 3 hr. with Girard's reagent D (*NN*-dimethylglycine hydrazide hydrochloride) (25 mg per g of extract), and the cooled soln. was mixed with an equal vol. of H_2O and decanted. A second extraction with an equal vol. of benzene removed all H_2O-insol. fractions. The aq. layer was shaken for 3 hr. with a soln. of dinitrophenylhydrazine equiv. to the wt. of reagent D, 2 ml of conc. HCl, 5 ml of ethanol, 15 ml of dichloromethane and 10 ml of H_2O. After being decanted, the solvent layer was washed with H_2O, dried over Na_2SO_4, and concentrated to 5 ml. The dinitrophenylhydrazones were roughly separated on a column of activated alumina with benzene and ethanol, and the yellow and orange fractions were concentrated to 2 ml and radially chromatographed in 5 ml of dimethylformamide, 4 ml of cyclohexane and 2 ml of cyclohexene. Chem. Abstr.

1597. **Study of the catalytic oxidation of propene. III. Methods for determining acraldehyde in the catalytic oxidation products of propene over a copper catalyst.** V. P. Latyshev and N. I. Popova. *Izv. Sibirsk. Otd. Akad. Nauk SSSR*, 1959, (9), 48–51; *Ref. Zhur., Khim.*, 1960, (12), Abstr. No. 47,226.—The bromide - bromate and spectrophotometric methods and the polarographic method of Kirillova and Korshunov (*Brit. Abstr. C*, 1952, 12) for determining acraldehyde (**I**) are compared. The bromide - bromate method is unsuitable; the spectrophotometric method gives low results in the presence of polymers of **I**; the polarographic method gives accurate results for the determination of **I** in all possible mixtures of the oxidation products of propene if 0·1N-HCl is used as basal electrolyte. Propionaldehyde, glyceraldehyde, acetaldehyde and pyruvaldehyde do not interfere. (*Cf.* Popova and Vermel', *Anal. Abstr.*, 1959, **6**, 599.) C. D. KOPKIN

1598. **Analysis of 1,2-glycols and polyhydric compounds. V. Determination of glyceraldehyde by means of the formaldehyde liberated with periodate.** L. Maros, I. Molnár-Perl and E. Schulek (Inst. Inorg. and Anal. Chem., L. Eötvös Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1960, **24** (1), 67–71 (in German).—The test soln. is set aside for 1 hr. with periodic acid soln. in a glass-stoppered conical flask. A slight excess of 1·5M-aq. Na_2SO_3 is added and the soln. is neutralised with 10% aq. acetic acid. A small excess of acid is added and the flask is closed and shaken. Pentane (5 ml) is added and the flask is set aside for 30 min.; a little starch soln. is added and the soln. is made alkaline with 20% NaOH soln.; 0·2 to 0·3 g of KCN is added and dissolved by swirling and after 2 to 3 min. the soln. is made slightly acid with HCl, and the liberated H_2SO_3 is titrated with 0·1N-iodine. An accuracy of ± 0·5% is claimed. The direct determination of glyceraldehyde with $NaHSO_3$ and standard iodine soln. is also described.
H. M.

1599. **Determination of acetals, ketals and vinyl ethers.** B. Budešínský and J. Körbl (Res. Inst. f. Pharm. and Biochem., Prague, Czechoslovakia). *Mikrochim. Acta*, 1960, (5–6), 697–702 (in German).—These substances are simultaneously hydrolysed and oximated in 30 min. at 100° by a soln. 1M in HBr and 0·1M in hydroxyammonium chloride. After cooling, the soln. is treated with 10M-KOH, and the excess of hydroxylamine is titrated potentiometrically with $K_3Fe(CN)_6$ soln. after 30 min. The potential at the end-point is ≃ − 200 mV. The accuracy on 20 to 150 mg is within ± 1·25%.
J. P. STERN

1600. **Combined detecting reagent for the identification of organic acids on paper chromatograms.** J. Pásková and V. Munk (Central Res. Inst. of Food Indust., Prague-Smichov, Czechoslovakia). *J. Chromatography*, 1960, **4** (3), 241–243.—The reagent consists of (*i*) a soln. containing 0·075% of bromocresol green and 0·025% of bromophenol blue in abs. ethanol and (*ii*) an aq. soln. containing 0·5% of $KMnO_4$ and 1% of $Na_2CO_3.10H_2O$. Equal volumes of (*i*) and (*ii*) are mixed and sprayed immediately. The mixed soln. is unstable after 10 min. Colours are tabulated for 18 common organic acids and enable identifications to be made of acids having similar R_F values; n-butanol - formic acid - water (10:1:5) is a suitable solvent.
R. M. S. HALL

1601. **Simplified chromatographic separation and analysis of C4 through C12 dibasic acids.** E. D. Smith (The Chemstrand Corp., Decatur, Ala. U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1301–1304.—*Procedure*—Mix coarse dry silicic acid (prep described) (25 g) with water or M-citrate buffer o the appropriate pH (16 ml) and pack with suctio in a column (40 cm × 2 cm). Add the sampl (≃ 50 mg) dissolved in $CHCl_3$ (10 ml) or in a 10% soln. of 2-methylbutan-2-ol in $CHCl_3$ (10 ml) an develop at a flow rate of 2 ml per min., by applica tion of pressure of N, with $CHCl_3$ followed b successive mixtures of $CHCl_3$ and n-butanol con taining increasing concn. of butanol up to 40% As stationary phases use H_2O to separate the C to C_7 acids, buffer of pH 5·40 for the C_7 to C_9 acids, and buffer of pH 7·8 for the C_9 to C_{12} acids Analyse the 10-ml fractions of eluate gravimetrically by evaporation and weighing. A precision of ± 5% is achieved. A. R. ROGERS

1602. **Micro-determination of chloro-substituted acetic acids.** F. Gölles (Inst. Pharmacognosy Univ. Graz, Austria). *Mikrochim. Acta*, 1960, (4) 597–606 (in German).—The method of Fischer and Kartnig (*Anal. Abstr.*, 1961, **8**, 1287) is applied to the rapid micro-determination of the three chloroacetic acids with phenol (critical mixing temp 65·5°). Mixture-gap diagrams are given and confirm the agreement of the max. temp. with those found on the Kofler block. The only other liquid suitable for use with all three chloroacetic acids is *m*-cresol (critical mixing temp. of 150°).
J. P. STERN

1603. **Determination of anhydrides of carboxylic acids by means of hexamethyleneimine and piperidine.** A. P. Terent'ev, S. I. Obtemperanskaya, M. M. Buzlanova and T. E. Vlasova. *Vestn. Moskov. Univ., Ser. Khim.*, 1960, II, No. 4, 71–73.—The method is based on the reaction of anhydrides with secondary amines and permits their determination in the presence of acid with an accuracy of ± 0·25%. *Procedure*—Place 0·05 g of anhydride in a 100-ml flask with a ground-glass stopper and add hexamethyleneimine or piperidine (0·1N in dioxan) (10 ml). After 5 min. add 5 drops of indicator, prepared by mixing 40 ml of 0·1% ethanolic methyl red, 10 ml of 0·1% ethanolic methylene blue, 50 ml of ethanol and 100 ml of water) and titrate with 0·1N-HCl in methanol to a colour change from green to pink.
A. BURWOOD-SMITH

1604. **Kinetic approach to the identification of aliphatic amines.** T. Aung, E. A. Healy and R. K. Murmann (Univ. of Missouri, Columbia, U.S.A.). *Chemist Analyst*, 1960, **49** (3), 73.—A primary aliphatic amine in the presence of H_2O, alcohols or secondary amines may be identified by measurement of the first-order dissociation in acid soln. of the complex between Fe^{II} and the Schiff base formed by the amine with picolinaldehyde (**I**). *Procedure*—To 0·1 ml of faintly acid soln. (≃ 0·2M in amine) add 0·1 ml of methanol and 1 drop of **I** - methanol soln. (1:3). Add 1 to 2 drops of 10% $FeCl_2$ soln. and shake. After 5 min. transfer 1 to 3 drops to 3 ml of 0·1 or 0·25M-HCl (adjusted with NaCl to an ionic strength of 1). Measure the change in extinction with time at a controlled temp.; the half-time (obtained from the graph of log extinction *vs.* time) is characteristic for each amine. Small amounts of aromatic amines do not interfere. P. D. PARR-RICHARD

1605. **Chromatography of organic substances. I. Determination of aliphatic amines by gas romatography.** J. Franc and M. Wurst (Forschgsinst. f. Org. Synthesen, Pardubice-Rybitví, echoslovakia). *Coll. Czech. Chem. Commun.*, 60, **25** (9), 2290–2295 (in German).—The amines e separated on a column of kieselguhr or Celite th a stationary phase of Elastomer E 301 (Griffin d George); N is used as the carrier gas and the erating temp. is 23°. Volatile amines are first nverted into hydrochlorides. The separated nines are burned in a combustion apparatus, the $_2$O formed being then converted into H over eated iron, and then determined by means of a ermal-conductivity cell. Relative elution times e given for 20 amines. The coeff. of variation r a single compound is ± 1·4% (100 determinaons). G. P. Cook

1606. **Analysis of hydroxamic acids and hydrzides: preparation and properties of dinitrophenyl erivatives of hydroxamic acids, oximes, hydrzides and hydrazones.** S. Seifter, P. M. Gallop, S. ichaels and E. Meilman (Albert Einstein Coll. of ed., Yeshiva Univ., New York 61, U.S.A.). *Biol. Chem.*, 1960, **235** (9), 2613–2618.—Three ethods are given for the determination of hydroamic acids. The first is a modification of Hill's ethod (*Brit. Abstr. C*, 1948, 257) and is used hen the excess of hydroxylamine has been moved by dialysis or extraction. The hydroxmic acid soln. is mixed with $Fe(ClO_4)_3$ soln. in 0% ethanol and the extinction is measured at 05 mμ. When hydroxylamine is present, the ethod of Lipmann and Tuttle (*Brit. Abstr. C*, 945, 276) is used. The second method is based n that of Bergmann and Segal (*Anal. Abstr.*, 956, **3**, 2122), which depends on the oxidation of ydroxamic acids by iodine to give nitrous acid, hich is then used to diazotise sulphanilic acid. he resulting diazonium compound is coupled with -naphthylamine, and the extinction is measured t 520 mμ. The third method depends on the eaction of free hydroxylamine with indole to give yellow compound, the extinction of which is easured at 400 mμ. In this method a preliminary ydrolysis of hydroxamic acid is needed to yield ree hydroxylamine. Amidoximes may also be etermined by any of these methods. When recautions are taken to ensure the removal of all ree hydroxylamine before the determination, the odine-oxidation and indole methods are much nore sensitive than the $Fe(ClO_4)_3$ method. An xtremely sensitive method is given for the deterination of hydrazides with *p*-dimethylaminoenzaldehyde. J. N. Ashley

1607. **Differentiation of some diazines.** B. Berisso (Univ. Nac. del Litoral, Rosario, Argenina). *Mikrochim. Acta*, 1960, (5–6), 898–901 (in panish).—The use of a 2% molybdophosphoric cid soln. for the identification and differentiation f piperazine, pyrazine, pyridazine and pyrimidine y means of the characters of the microcrystalline pt. formed is described. N. E.

1608. **Amperometric titration of EDTA and DCyTA.** D. Roubalova and J. Doležal (Pedagogic High School, Charles' Univ., Prague, Czechoslovakia). *Chemist Analyst*, 1960, **49** (3), 76.—EDTA and DCyTA (1,2-diaminocyclohexane-*NNN'N'*-tetra-acetic acid) (**I**) soln. can be titrated mperometrically with $Hg(NO_3)_2$ soln. in a mixture f 0·2M-Na acetate and 0·2M-acetic acid at pH 5 to 6, and an applied potential of 0·1 V; fuchsine is used as a max. suppressor. The average error was 0·2%. Determination of Hg^{II} by titration with EDTA or **I** was also successful. P. D. Parr-Richard

1609. **Analysis of sulphonic acids and salts by gas chromatography of volatile derivatives.** J. J. Kirkland (Ind. and Biochem. Dept., Exp. Sta., E.I. du Pont de Nemours and Co., Inc., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1388–1393.—The quant. conversion of the acids and salts into volatile derivatives, and the subsequent analysis, with thermistor detectors, are described. Aliphatic, aromatic and alkylarylsulphonic acids and their salts are converted into their corresponding sulphonyl chlorides by the use of thionyl chloride or phosgene in the presence of a formamide derivative as a catalyst. Free sulphonic acids are converted into their corresponding methyl esters by the use of diazomethane, the preparation of which is described. Recoveries of 96 to 100% are attained for all reactions carried out by the recommended procedures. C. B. Baines

1610. **New colorimetric applications of the chromate - o-dianisidine system. II. Indirect colorimetric determination of some mercapto-compounds: thioglycollic acid, thiolactic [2-mercaptopropionic] acid and thiomalic [mercaptosuccinic] acid.** F. Buscaróns, J. Artigas and C. Rodriguez-Roda (Fac. of Sci., Univ., Barcelona, Spain). *Anal. Chim. Acta*, 1960, **23** (3), 217–218 (in English).—The compounds named are treated with an excess of K_2CrO_4 and the excess is determined by measuring the red colour produced when the K_2CrO_4 reacts with *o*-dianisidine in acid soln. *Procedure*—Potassium chromate soln. (200 μg per ml) (2 ml) is transferred to a 100-ml flask and the test soln. is added from a micro-burette, in an inert atmosphere to avoid oxidation of the mercapto-compounds; 2 to 3 ml of 5N-H_2SO_4 is added and the soln. is set aside for 5 to 10 min. *o*-Dianisidine soln. (0·5 g dissolved in 50 ml of acetone and diluted to 100 ml with water) (0·5 ml) is added and the red colour is measured at 470 mμ, 5 to 25 min. later. Results are referred to the appropriate calibration curve. Low concn. (0·1 to 5 μg per ml) can be determined. H. M.

1611. **Differential conductimetric titration of a mixture of methylchlorosilanes in non-aqueous solutions.** A. P. Kreshkov and V. A. Drozdov. *Dokl. Akad. Nauk SSSR*, 1960, **131** (6), 1345–1348; *Ref. Zhur., Khim.*, 1960, (18), Abstr. No. 73,199.—The determination of di- and tri-component mixtures of $(CH_3)_3SiCl$, $(CH_3)_2SiCl_2$ and CH_3SiCl_3 is based on the quant. conversion of the chlorosilanes into the alkylthiocyanato-derivatives by the action of NH_4SCN, and conductimetric titration in methyl cyanide - ethyl ether (2:3) with 0·1M-amidopyrine in benzene. The mono-substituted compound is first titrated, then the di-substituted, and finally the tri-substituted. The smallest amounts which can be determined in the mixture are, $(CH_3)_3SiCl$ 0·0055 g, $(CH_3)_2SiCl_2$ 0·0025 g, and CH_3SiCl_3 0·0023 g in 12 ml. Concentrated soln. must be diluted. The error in determining the individual chlorosilanes is ± 2%. C. D. Kopkin

1612. **Potentiometric titration of individual alkylchlorosilanes with organic bases in methyl cyanide.** A. P. Kreshkov, V. A. Drozdov and E. G. Vlasova. *Izv. Vŷssh. Ucheb. Zavedeniĭ, Khim. i Khim.*

Tekhnol., 1960, **3** (1), 85–87; *Ref. Zhur., Khim.*, 1960, (18), Abstr. No. 73,200.—Titration in methyl cyanide with 0·05N-phenazone or nitron in methyl cyanide, or with a soln. of amidopyrine in benzene, by visual and potentiometric methods is suggested. The indicators used were crystal violet, dimethylaminoazobenzene, bromocresol purple, methyl orange, bromophenol blue and gallo sea blue (C.I. Mordant Blue 14); $(CH_3)_3SiCl$, $(CH_3)_2SiCl_2$, CH_3SiCl_3 and $SiCl_4$ were titrated; the error of the determination is $\pm$ 0·3%. The addition of benzene, toluene, chlorobenzene or CCl_4 does not affect the results. A new cell is described for potentiometric titrations in non-aq. solvents of low dielectric constant. C. D. KOPKIN

1613. **Potentiometric titration of nitrogen-containing organosilicon compounds in non-aqueous media.** A. P. Kreshkov, V. A. Drozdov and E. G. Vlasova. *Izv. Vŷssh. Ucheb. Zavedenič, Khim. i Khim. Tekhnol.*, 1960, **3** (1), 80–84; *Ref. Zhur., Khim.*, 1960, (18), Abstr. No. 73,203.—It is suggested that nitrogen-containing organosilicon compounds be titrated with $HClO_4$ soln. in methyl cyanide, methyl nitrite, or their mixtures with benzene or dioxan, by visual or potentiometric methods. The indicators used were crystal violet, bromocresol purple, bromophenol blue, cresol red, dimethylaminoazobenzene, thymol blue, methyl red and methyl orange. Compounds containing N attached directly to Si, and compounds with N in an organic radical, have been titrated. The potential jumps when titrating in methyl nitrite are 10 mV larger than those in methyl cyanide. The amount of titrating soln. used is proportional to the number of N atoms in the molecule of the compound. The error is $\gtrsim$ 1·5%. C. D. KOPKIN

1614. **Analysis of dilute solutions of triethylaluminium [in petroleum spirit].** R. Z. Lioznova and M. L. Genusov (Okhtensk Chem. Combine). *Zavod. Lab.*, 1960, **26** (8), 945–947.—Apparatus for determining $(C_2H_5)_3Al$ from the volume of ethane liberated by the action of water is described. A correction is made for the solubility of ethane in the solvent. G. S. SMITH

1615. **Partition chromatography of aromatic hydrocarbons on rubber-like polymers.** G. L. Starobinets and E. V. Povarkov (Lenin Univ., Minsk, USSR). *Zhur. Anal. Khim.*, 1960, **15** (4), 405–408.—In this chromatographic method the carrier of the stationary phase is omitted and the components to be separated are distributed between the stationary high-molecular-weight phase [a vulcanisate of unfilled polymer SKB (sp. gr. 1·002) containing 2% of S] and a low-molecular-weight mobile phase (methanol - H_2O, containing from 24·6 to 37·2% by vol. of H_2O depending on the coeff. of distribution of the hydrocarbons between the polymer and the methanol - H_2O). Chromatography is carried out in a column of diameter 1·6 cm with a 26·4-cm layer of stationary phase. Part of the stationary phase is removed from the column and then the test mixture is introduced directly into the upper part of the stationary phase. After adsorption for 1 to 2 hr., the mobile solvent is passed through at the rate of 0·2 ml per min. A typical mixture separated contained benzene (0·1 ml), toluene (0·15 ml), *p*-xylene (0·2 ml) and mesitylene (0·1 ml). The amounts of these hydrocarbons recovered were, respectively, 0·105, 0·155, 0·21 and 0·1 ml. The mean error is $\gtrsim$ 5%. K. R. COOK

1616. **Fluorescence spectra of aromatic hydrocarbons and heterocyclic aromatic compounds.** B. L. Van Duuren (Inst. of Ind. Med., New York Univ. Med. Centre, U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1436–1442.—The application of fluorescence spectroscopy to the qual. and quant. analysis of aromatic compounds is described. The effects of concn., solvent and pH on fluorescence intensity are examined for a number of compounds of differing fluorescent properties. The effects of replacing hydrogen atoms of aromatic hydrocarbons by alkyl groups, and of carbon atoms by nitrogen and oxygen, are also examined. Results obtained augment identifications of compounds made on the basis of u.v. and fluorescence emission spectra. C. B. BAINES

1617. **Determination of benzene, toluene and xylene in their mixtures by gas - liquid chromatography.** D. A. Vyakhirev and M. I. Ostasheva *Trudŷ Khim. i Khim. Tekhnol.*, 1959, (1), 128–132; *Ref. Zhur., Khim.*, 1960, (16), Abstr. No. 65,185.—The separation and determination of mixtures of benzene, toluene and xylene are carried out at 100° in a stream of N (80 ml per min.) on a column (100 cm long, 0·6 cm internal diam.) of diatomite containing 30% (w/w) of triethylene glycol. Results calculated from the peak heights were more accurate than those calculated from the peak areas. The errors are $< 5\%$. K. R. COOK

1618. **Chromatographic determination of the content of heterocyclic sulphur compounds in commercial benzene and naphthalene.** M. Hřivnáč and J. Janák (Lab. Gas Analysis, Brno, Czechoslovakia). *Chem. Průmysl*, 1960, **10** (8), 399–403.—The analysis was carried out in a Griffin and George apparatus, type MK II, with polyoxyalkylene adipate (Reoplex 400, Geigy) (30%) as stationary phase, on Celite 545 for thiophen separation (temp. 85°) or on Sterchamol (Dortmund) for thionaphthen separation (temp. 175°). Hydrogen was used as carrier gas. *Determination of thiophen in benzene by the standard addition method*—Place 40 to 100 μl of the sample on the column and chromatograph at 85°. Repeat the procedure with the same sample to which has been added 10 μg of thiophen per ml. *Determination of thionaphthen in technical naphthalene by the internal standard method*—Melt the sample (5 g), stir to homogenise and cool. Into a small test-tube weigh durene (1,2,4,5-tetramethylbenzene) (10 mg) and cover it with the test sample (1 g). Add $\simeq$ 5 drops of 1-methyl-naphthalene to decrease the m.p. Heat till just liquid, place 40 to 100 μl on the column and chromatograph at 175°. Samples containing $>$ 0·05% of thiophen or $>$ 0·1% of thionaphthen can be analysed within 40 min. with an accuracy of $\pm$ 10%. J. ZÝKA

1619. **Determination of ethylbenzenes.** D. E. Nicholson (Humble Oil & Refining Co., Baytown, Tex.). *Anal. Chem.*, 1960, **32** (10), 1372.—Infrared data are given for ethylbenzene and the three diethylbenzenes.

1620. **Analysis of phenylalkanes by gas chromatography.** C. F. Spencer and J. F. Johnson (California Research Corp., Richmond, U.S.A.). *J. Chromatography*, 1960, **4** (3), 244–248.—Retention times are listed for the series phenylhexanes to phenyleicosanes and some n-paraffin reference compounds from n-decane to n-tetracosane with a 12-ft. asphalt - firebrick column at 240°, 270°

320°. Separation of some isomers is not ays possible. Accuracies given for the deter- ation of 1- to 6-phenyldodecane mixtures range n + 5 to − 8%. R. M. S. Hall

621. **Gas - liquid chromatographic separation and lysis of chlorinated toluenes.** S. K. Freeman nzol Products Co., Newark, N.J., U.S.A.). *al. Chem.*, 1960, **32** (10), 1304–1306.—To separate analyse a mixture of ring and side-chain Cl vatives of toluene, use a 7-ft. column of 20% ilicone oil (Dow Corning No. 550) on firebrick rrell, Kromat-FB) at 170° in combination with -ft. column of 20% of Ucon 2000 on firebrick 170°, with He as carrier gas at a flow rate of ml per min. Peak-area correction factors for ixture of 7 components ranged from 0·73 to 1·55. A. R. Rogers

622. **Quantitative determination of the main urities in technical cumene hydroperoxide by a-red absorption spectra.** N. K. Rudnevskiĭ . V. V. Zharkov. *Trudy Khim. i Khim. Tekhnol.*, 9, (1), 119–124; *Ref. Zhur., Khim.*, 1960, (18), str. No. 73,191.—A method is developed for the nt. determination of cumene hydroperoxide (**I**), ethylphenylcarbinol (**II**) and acetophenone (**III**) he products of the oxidation of cumene (**IV**), by ir i.r. absorption spectra. **III** is determined ependently of the content of the other com- ents by measuring the extinction by the base- e method at 5·93 μ. **I** is also determined inde- ndently at 11·98 μ, on the assumption that the xture of **I** and **IV** is a binary mixture, because of low content of **II** and **III**. **II** is determined at 55 μ by the method of successive approxima- ns, allowing for the concn. of **I**. The relative ors are, for **I**, ± 1·6% (concn. range 19 to 86%); **II**, ± 5·5% (1 to 4%); and for **III**, ±5% 5 to 2·5%). C. D. Kopkin

623. **Analysis of multicomponent mixtures by ra-red absorption spectra. II. Determination of orocumene, t-butylchlorobenzene and chloro- henylmethane isomers.** Yu. Ya. Mikhaĭlenko, N. Lebedev, I. K. Kolchin and E. G. Kutyrina endeleev Chem.-Technol. Inst., Moscow). *Zhur. al. Khim.*, 1960, **15** (4), 495–499.—Sylvite cells used in the spectrophotometer with a path gth of 0·09 cm, the method being that described eviously (*Anal. Abstr.*, 1960, **7**, 5295). Suitable velengths for *o-*, *m-* and *p*-chlorocumene are 27, 12·77 and 12·11 μ, respectively; for *o-*, *m-* d *p*-t-butylchlorobenzene, 13·03, 12·75 and 12·10 μ, pectively; and for *o-*, *m-* and *p*-chlorodiphenyl- thane, 13·35, 12·95 and 12·63 μ, respectively. x. absorption occurs at 9·64 μ and at 9·09 μ. e extinction is measured in CS_2 soln. Deter- nations of synthetic mixtures of the compounds ntioned have a mean relative error of $\simeq$ 4%. K. R. Cook

624. **Determination of o-, *m*- and *p*-cresols m their infra-red absorption spectra.** N. A. umaevskiĭ (Inst. of Elemento-organic Compounds, ad. Sci., USSR). *Zavod. Lab.*, 1960, **26** (8), 957–9.—Contents are determined from measurements absorption at 756, 778 and 816 cm^{-1}. G. S. Smith

625. **Spot-test detection and colorimetric deter- nation of aliphatic aldehydes with 2-hydrazino- nzothiazole. Application to air pollution.** E. wicki and T. R. Hauser (Air Pollution Engng Res., Robert A. Taft Engng Center, U.S. Dept. of Health, Educ. and Welfare, Cincinnati, Ohio). *Anal. Chem.*, 1960, **32** (11), 1434–1436.—The test described is especially sensitive to formaldehyde. The sample of vapour or soln. is brought into contact with a soln. of 2-hydrazinobenzothiazole in HCl, and $K_3Fe(CN)_6$ soln. is added to give a brown colour, which changes to purple on addition of NaOH soln. and to an intense blue on subsequent addition of dimethylformamide. Procedures are described for spot tests on plates or paper and on silica gel contained in a tube, and for a colorimetric determination. Identification limits for formalde- hyde, acetaldehyde and propionaldehyde are 0·01, 0·3 and 0·5 μg, respectively, but are much higher for other aliphatic aldehydes. Aromatic and heterocyclic aldehydes give negative results. High concn. of nitromethane interfere. G. Burger

1626. **Determination of aromatic aldehydes by near-infra-red spectrophotometry.** R. M. Powers, J. L. Harper and Han Tai (A. E. Staley Mfg. Co., Decatur, Ill., U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1287–1289.—The method described can be used to determine aromatic aldehydes in the presence of aromatic ketones. Of three charac- teristic absorption bands, the 2·21-μ region is used because it is relatively free from interference, the only interferences in this region being from terminal epoxides and cyclopropyl groups. K. A. Proctor

1627. **Mass spectra of aromatic esters.** E. M. Emery (Colgate-Palmolive Co., Jersey City, U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1495–1506.—The mass spectra are listed of 25 aliphatic esters of aromatic acids and 17 aromatic esters of aliphatic acids. Characterisation of these esters is obtained from fragmentation patterns and the spectra can be used for the identification of unknown esters and the determination of esters in mixtures. Examples are given of several analyses of synthetic mixtures showing excellent results. C. B. Baines

1628. **Non-aqueous titration of phenolic esters.** B. Smith and Å. Haglund (Inst. f. Organsk Kemi, Chalmers Tekn. Högskola, Göteborg, Sweden). *Acta Chem. Scand.*, 1960, **14** (6), 1349–1352.—Potentiometric titration of phenolic esters with 0·1N-tetra-n-butylammonium hydroxide in benzene - methanol as titrant and acetone as solvent is a suitable method for differentiating between various types of ester. Results are tabulated for 27 phenolic esters. The esters of aliphatic carboxylic acids can be titrated provided that strongly electron-repelling groups are not present in the phenolic ring. Un- substituted esters of aromatic carboxylic acids do not react, but the introduction of an electron- attracting group into the aromatic nucleus can make such esters reactive. The mechanism of the reaction is briefly discussed, together with some exceptions to the general rules. S. M. Marsh

1629. **Effect of substituent groupings on the chromatographic behaviour of phenoxyacetic acids. I. Methyl-substituted phenoxyacetic acids.** L. S. Bark and R. J. T. Graham (Roy. Tech. Coll., Salford, England). *Analyst*, 1960, **85**, 663–666.—An investigation by paper chromatography of a series of methyl-substituted phenoxyacetic acids suggested that the phenoxyacetate group plays a dominant part in the adsorption of the acids or their ammonium salts on material with which the carboxyl group can form hydrogen bonds, and is

therefore the fundamental group. To ensure that the variation in the R_F and R_M values of the series was produced only by changes in the chemical structure, the conditions were rigorously standardised. The solvents were n-butanol saturated with water and n-butanol saturated with aq. NH_3. The results suggest that, in addition to the fundamental group factors, there are, for these nuclear-substituted compounds, at least two factors dependent on the position of the steric group relative to the fundamental group, and on the position of the substituent groups relative to each other, and that the greater the distance between the fundamental group and the substituent group the smaller the effect on the R_M value of the molecule, and that the nearer the substituent groups are to one another the smaller is the effect on the R_M value.

A. O. JONES

1630. **Determination of aromatic primary amines and compounds containing reactive methylene groups by diazotisation and C-nitrosation reaction using alkyl nitrites.** N. K. Mathur, S. P. Rao and Dau Narain (Jaswant Coll., Jodhpur, India). *Anal. Chim. Acta*, 1960, **23** (4), 312–316 (in English). —Primary amines, acetylacetone and dimedone in HCl soln. (4N) may be titrated potentiometrically with alkyl nitrite soln. The temp. should be kept between 5° and 10°. T. R. ANDREW

1631. **Solvent and concentration effects on the near-infra-red N–H bands of primary aromatic amines.** K. B. Whetsel, W. E. Roberson and M. W. Krell (Tennessee Eastman Co., Div. of Eastman Kodak Co., Kingsport, U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1281–1286.—The effects of change of concn. in the range 0·5 to 100% and change of solvent on the first overtone stretching and combination N–H bands of aniline, *m*-toluidine, *o*-chloroaniline and *m*-chloroaniline were determined in the solvents cyclohexane, CCl_4, CS_2, benzene, $CHCl_3$ and methyl cyanide. Quant. analysis in the overtone region can be made in 10-cm cells at concn. $< 0{\cdot}1$M in all solvents except cyclohexane, but only CCl_4, CS_2 and $CHCl_3$ can be used in the combination region. Deviations from Beer's law are serious in 1-cm cells at concn. between 0·1M and M in cyclohexane, CS_2 and CCl_4. An improvement in accuracy is possible by using integrated molar extinction coeff.

A. R. ROGERS

1632. **Ultra-violet spectrophotometric analysis of solutions of benzonitrile and benzamide.** M. J. Astle and J. B. Pierce (Case Inst. of Technol., Cleveland, Ohio, U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1322–1324.—The molar fractions of benzonitrile (**I**) and benzamide (**II**) in soln. in the presence of water and alcohols can be determined by measurement of the u.v. absorption at a single wavelength if the total number of moles of **I** and **II** is held constant, and the molar fraction is used as the unit of concn. A. R. ROGERS

1633. **[Infra-red] spectrometric analysis of the isomeric composition of mixtures of diamino- and di-isocyanato-toluenes.** A. I. Finkel'shteĭn and E. N. Boĭtsov. *Zavod. Lab.*, 1960, **26** (8), 959–963.—With nitromethane as solvent, 2,4- and 2,6-diaminotoluenes are determined from infra-red absorptions at 11·86, 12·57 and 12·87 μ, and 2,4- and 2,6-di-isocyanatotoluenes are determined from absorptions at 12·35 and 12·80 μ.

G. S. SMITH

1634. **Titanous chloride as a reagent for quan tative organic micro-analysis. II. Micro-determin tion of azo and diazonium compounds and nitroarylhydrazines.** J. V. Earley and T. S. M (Chem. Dept., Brooklyn Coll., N.Y., U.S.A *Mikrochim. Acta*, 1960, (5–6), 685–692 (in Englis —Azo and diazonium compounds and nitroary hydrazines are determined in acetate buffer room temp. by reaction with excess of $TiCl_3$ sol and back-titration with $(NH_4)_2SO_4.Fe_2(SO_4)_3$. T accuracy on 4 to 8 mg of sample is within $\pm$ 0·2% Titration with $TiCl_3$ in strong HCl permits t determination of many azo and nitroso compoun in the presence of many nitro compounds (*cf.* M and Earley, *Anal. Abstr.*, 1959, **6**, 3558).

J. P. STERN

1635. **A new spot-test reaction for various nitr benzene derivatives.** V. Anger (Forschungsla der Firma Loba Chemie, Wien XIX, Austria *Mikrochim. Acta*, 1960, (5–6), 827–830 (in German —Diphenylbenzidine (**I**) in 80% H_2SO_4 is oxidise by nitroso-compounds; certain aromatic nitr compounds give a similar reaction after reductio with Devarda's alloy. Possible reaction mechanism are discussed. *Procedure*—To the aq. or ethanol soln. of the sample in a clean test-tube add 0·5 m of **I** soln. (1 mg of **I** in 10 ml of 80% H_2SO_4) an 1 drop of reducing agent (10 mg of powdered allo suspended in 10 ml of H_2O) and warm at 100 A positive reaction is shown by the appearance a blue colour after 1 to 5 min.; the limit of dete tion is 5 to 10 μg. It is possible to distinguis between, *e.g.*, *p*-chloronitrobenzene (positive re action) and *o*-chloronitrobenzene (negative). Nitro benzene and its homologues do not react.

P. D. PARR-RICHARD

1636. **Qualitative test for mononitrotoluenes wit trisodium pentacaynoamminoferrate.** C. B. Hacket and R. M. Clark (Res. and Devel. Dept., Nob Div., I.C.I. Ltd., Stevenston, Ayrshire, Gt. Britain *Analyst*, 1960, **85**, 683–684.—A drop of the sampl or 10 mg of the sample in hot ethanol (3 ml) i treated with 7 drops of 10% aq. $CaCl_2$ and the with 50 mg of zinc dust. The mixture is heated i a water bath, boiled for 1 min., filtered and cooled Four drops of the filtrate are diluted to 2 ml and drop of freshly prepared 1% aq. trisodium penta cyanoamminoferrate soln. is added. *o*-Nitrotoluen forms a pink or red colour, *m*-nitrotoluene a reddish violet and *p*-nitrotoluene a violet - blue colou The reaction does not occur in the absence of ligh Nitrobenzene, 2,4-dinitrotoluene and 2,4,6-trinitro toluene interfere with the test by formin reddish-violet colours. Nitrobenzene is not normall present in commercial explosives; the other tw interfering compounds can be distinguished b Janowski's reaction. A. O. JONES

1637. **Identification of adsorbed species by infra red spectroscopy. Thiophen - cobalt molybdat systems.** D. E. Nicholson (Res. and Devel. Div Humble Oil & Refining Co., Baytown, Tex U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1365–1366.— Several possibilities for the structure of adsorbe thiophen are suggested by i.r. studies.

K. A. PROCTOR

1638. **New specific analytical procedures for th detection and characterisation of 1,4-naphtha quinones.** E. Sawicki and W. C. Elbert (R. A Taft San. Engng Center, U.S. Dept. of Health Educ. and Welfare, Cincinnati, Ohio). *Anal*

m. Acta, 1960, **23** (3), 205–208 (in English).—tain derivatives of 1,4-naphthaquinone, *viz*, the ıethyl-, 2-chloro-, 2,3-dichloro- and 2,3-dibromo-ıpounds, give a red colour when treated with minothiophenol in dil. HCl. The colour changes blue with conc. HCl. Derivatives with an ctron-donor group in the 2-position give negative ılts, as do other quinones. *Procedure*—Place a p of reagent soln. (0·5 ml of 2-aminothiophenol l 4 ml of conc. HCl, diluted to 50 ml with water) a filter-paper, followed by 1 μl of the methanolic c soln. in the centre of the drop. After a few ıutes note the colour, then add a drop of conc. l and again note the colour. The identification its are in all cases $< 0{\cdot}5$ μg. Compounds ing a positive test may be differentiated by .mination of their absorption spectra in acid ı. 2-Hydroxy-1,4-naphthaquinone (**I**) may also detected by the following procedure. Place a p of the reagent soln. (0·5 g of *o*-phenylene-mine dissolved in 4 ml of conc. HCl and 20 ml water and then diluted to 50 ml with water) a filter-paper, followed by 1 μl of the test soln. acetic acid. Heat the spot in a jet of steam for min., add a drop of 10% aq. KOH soln. and .mine under u.v. light. A bright rose-red ɔrescence indicates the presence of **I**. The ntification limit is 0·3 μg. H. M.

1639. **Simple spot test for pyrene and its derivatives: application to air-pollution studies.** E. vicki and T. W. Stanley (R. A. Taft San. Engng ıter, U.S. Dept. of Health, Education and elfare, Cincinnati, Ohio). *Chemist Analyst*, 1960, (3), 77–78.—*Procedure*—Evaporate the soln. to rness, dissolve the residue in 0·1 ml of acetic d - fuming HNO_3 (1:1) and heat at 100° for 5 ı. Evaporate to dryness and add 1 drop of ıethylformamide. Place 1 drop of 25% aq. raethylammonium hydroxide soln. on filter-ɔer and apply 1 μl of test soln. to the centre. rene and certain derivatives give a blue colour; ntification limits range from 0·01 to 10 μg. veral common polynuclear hydrocarbons do not erfere. P. D. Parr-Richard

1640. **Determination of piperazine as piperazine cetate.** G. R. Bond, jun. (Houdry Process ɔp., Marcus Hook, Pa., U.S.A.). *Anal. Chem.*, 50, **32** (10), 1332–1334.—*Procedure*—Melt the mple in a closed container at the minimum temp. d stir. Dissolve a portion (1 to 3 g) in acid-free etone, dilute to 100 ml and mix. Dilute a 25-ml quot with acetone (25 ml), add an excess of cial acetic acid (0·35 to 1·05 ml), stir well, set de for 5 min. and filter through sintered glass. y the ppt. *in vacuo* over H_2SO_4 and weigh. The ɔerazine content of crude reaction mixtures over e range 1 to 100% can be determined with an curacy of $\pm$ 0·2% absolute in the absence of ɔylpiperazines, which are co-precipitated. Small ıounts of water in the acetone do not interfere. A. R. Rogers

1641. **High-resolution mass spectrometry. Interɔetation of spectra of petroleum fractions.** E. G. rlson, G. T. Paulissen, R. H. Hunt and M. J. Neal, jun. (Houston Res. Lab., Shell Oil Co., xas, U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1489–94.—The instrument described is a double-using, coincident-field, mass spectrometer which s a resolving power approaching one order of gnitude higher than that of the conventional alytical instrument, and has been adapted for operation with high-boiling petroleum fractions New techniques are developed for petroleum components, and new molecular-type fractions are established. Applications, primarily with the mass region below *m/e* 400, are described for pure hydrocarbons, pure compounds of high molecular weight, carbon-number distribution of sulphur types, alkyl thiophens, non-hydrocarbon components in catalytic cracking streams and microcrystalline wax. C. B. Baines

1642. **Rapid chemical determination of iron, nickel and vanadium in petroleum oils.** J. S. Forrester and J. L. Jones (Esso Res. Lab., Humble Oil & Refining Co., Baton Rouge, La., U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1443–1446.—Low-temp. wet-ashing with HNO_3 - $HClO_4$, followed by chelation with 4,7-diphenyl-1,10-phenanthroline (**I**), furil α-dioxime (**II**) and 3,3′-dimethylnaphthidine (**III**) permits the rapid colorimetric determination of Fe, Ni and V, respectively, in petroleum oils. *Procedure*—To the oil (0·2 to 2 g) add satd. NaCl soln. (2 ml), HNO_3 (20 ml) and $HClO_4$ (15 ml) and heat until the exothermic reaction starts. Stop heating, then re-heat at intervals until the vol. is about 6 ml. Add H_2O (10 ml), boil for 1 min., cool and dilute to 25 ml. To determine Fe, add hydroxyammonium chloride soln. (10%) (2 ml) and Na acetate soln. (10%) (8 ml) to 5 ml of this soln., adjust to pH 3 to 4 with 20% NaOH soln., add 4 ml of **I** soln. (0·001M in ethanol) and extract with isoamyl alcohol (6 ml). Dilute the extract to 25 ml with ethanol and measure the extinction at 533 mμ in a 1-cm cell against a reagent blank. Calculate the Fe content from a calibration curve. To determine Ni, add 10% Na citrate soln. (5 ml), phenolphthalein (5 drops) and conc. aq. NH_3 (3 drops) to 10 ml of the original soln. Neutralise with 20% NaOH soln. and add 3 drops in excess. Add 3 ml of **II** soln. (1% in 50% ethanol) and extract with $CHCl_3$ (2 $\times$ 10 ml). Dilute the extract to 25 ml with $CHCl_3$, add 1 g of Na_2SO_3 and determine the extinction at 435 mμ in a 1-cm cell against a reagent blank. To determine V, add H_3PO_4 (2·5 ml) and **III** soln. (0·1% in glacial acetic acid) (2·5 ml) to 10 ml of the sample soln. and dilute to 25 ml. After 15 min. measure the extinction at 550 mμ against a reagent blank. J. P. Stern

1643. **Determination of halogens in polymers and other petroleum products by a two-furnace combustion method.** E. W. Seefield and J. W. Robinson (Esso Standard Div., Humble Oil and Refining Co., Baton Rouge, La., U.S.A.). *Anal. Chim. Acta*, 1960, **23** (4), 301–305 (in English).—The problem of complete combustion of samples greater than 0·2 g has been overcome by the use of a furnace tube heated to 500° in one section and to 1000° in a second section. The sample (up to 20 g) is ignited in a stream of O (3 litres per min.) at 500° until combustion appears to be complete and is then pushed into the hotter zone for 10 min. The evolved gases are absorbed in a soln. containing NaOH (2%) and hydrazine sulphate (0·5%) and titrated potentiometrically with $AgNO_3$ (0·01N). T. R. Andrew

1644. **Rapid combustion method for determination of phosphorus in petroleum products.** S. J. Gedansky, J. E. Bowen and O. I. Milner (Res. Dept., Socony Mobil Oil Co., Inc., Paulsboro, N.J., U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1447–1449.—A method is described in which the sample is mixed with anhydrous Na_2CO_3 before it is burned

in O in a closed flask. The phosphate that is formed is dissolved in dil. H_2SO_4 and can then be determined by the hydrazine - ammonium molybdate method. If much Ba is present the soln. may be centrifuged just before the colour measurement, but this is not usually necessary. The procedure is suitable for lubricating oils and additives. A sample of < 50 mg is required, containing between 0·013 and 0·4 mg of P. The precision (standard deviation = 0·0016 for low concn. and 0·028 for high concn. of P) is equal to that of the ZnO method. The time required for one analysis is 20 min.

G. BURGER

1645. **Analytical separation of asphaltic constituents.** J. Buchta. *Ropa a Uhlie*, 1960, **2** (10), 268–269 (in Czech.).—The analytical separation of asphaltic constituents by chromatography on active alumina is described. Asphalt dissolved in CCl_4, after being fixed on the adsorbent, is eluted successively with CCl_4 (oil constituents), benzene (oil resins) and $CHCl_3$ (soft asphalts). The amount of asphaltenes is calculated by difference. Because of the efficiency of the chromatographic separation, it is suggested that this procedure should be used in preference to the determination of asphaltenes by hexane. FUEL ABSTR.

1646. **Elemental interaction effects in the spectrochemical determination of additive elements in lubricating oils.** E. L. Gunn (Humble Oil and Refining Co., Baytown, Tex., U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1449–1455.—A study of the interaction of Ca, Ba, P and Zn has been made in which a statistical design was employed. The direction and magnitude of interaction effects are well defined, and correction procedures are given. The effects of additive type, physical properties of the oil and instrumental variations are also considered, including a comparison of the porous-cup and rotating-disc techniques. C. B. BAINES

1647. **Determination of total water in coal.** D. J. W. Kreulen (Lab. D. J. W. Kreulen, Rotterdam, Netherlands). *Chem. Weekbl.*, 1960, **56** (40), 548–549.—The determination of total water in coal containing inherent water is discussed. During evaporation of the water, the temperature of the coal may in some cases be so much decreased that the prescribed minimum temperature cannot be maintained, so that a low water content is obtained. In such cases it is proposed to dry the coal in air before drying in an oven, and to increase the drying time to 1 hr. M. J. MAURICE

1648. **The use of partial demineralisation in coal analysis.** D. L. Ward and J. O'N. Millott (Nat. Coal Board, Coal Survey Lab., 20 Dee Hills Park, Chester, England). *Fuel, London*, 1960, **39** (4), 293–300.—Nearly all the mineral matter, except pyrites, is removed from coal by extraction with aq. HCl, aq. HF and conc. HCl, successively. The residue is washed with H_2O and dried *in vacuo*; C, H and calorific value are determined on the residue. A correction for absorbed HCl is obtained by determination of Cl in the coal, and for pyrites by determination of Fe in the ash. The corrected values are reported as based on the dry, ash-free coal. For coals with high ash content the results differ from those obtained by the normal method, based on mineral matter calculated by standard formulae, and are probably more accurate. The determination of volatile matter by this method gave variable results. Further investigation is needed. G. BURGER

1649. **Rapid method for the quantitative dete mination of phosphorus in coal.** M. V. Voĭnalovi and G. V. Sipovskiĭ. *Dokl. Akad. Nauk Tadzh.SS* 1959, **2** (3), 11–15; *Ref. Zhur., Khim.*, 1960, (1 Abstr. No. 47,114.—The photometric meth described is based on the formation of molybdenu blue by reduction with hydrazine sulphate, a can be used to determine 3 to 70 μg of P. Fer iron is first reduced to Fe^{2+} by Na_2SO_3 in ac medium. A correction must be made for t content of P in the reagents. Five parallel dete minations can be carried out in a working da The method is simple and rapid as compared wi the titrimetric method, and gives reproducib results. The error in determining 0·065 to 0·5 of P is 0·01 to 0·02% absolute, and 2 to 3% relativ C. D. KOPKIN

1650. **Analysis of metol - quinol developers paper chromatography.** J. Šimek (Electrotec Fac., ČVUT, Prague). *Chem. Průmysl*, 1960, (8), 403–407.—The quant. determination of met and quinol and their reaction products with alkali Na_2SO_3 has been studied chromatographicall *Procedure*—Dilute the developer (50 ml) wi glacial acetic acid (20 ml), place a suitable amou on Whatman No. 4 paper and treat by t ascending technique with n-butanol - acetic acid H_2O (14:2:5), n-butanol - formic acid (85%) - H_2 (18:2:5) or ethyl acetate - acetic acid - H_2O (40:7: as solvent, detect the separated spots by sprayin with a 3% soln. of $AgNO_3$ in aq. NH_3. Cut o the corresponding strips of the main paper, transf to a titration vessel, dilute with H_2O (100 ml), ad acetate buffer soln. (300 g of cryst. Na acetat 900 ml of H_2O and 1 ml of glacial acetic acid) an an excess of 0·01N-iodine and after 30 sec. bac titrate with $Na_2S_2O_3$ soln., with starch as indicato Quinol-mono- and -di-sulphonic acids, which a formed in developers by the action of alkali Na_2SO_3, are determined by the same method, t back-titration being carried out after 10 min.

J. ZÝKA

1651. **A modern approach to the evaluation bergamot oil.** F. C. Theile, D. E. Dean and Suffis (Shulton Inc., 630, 5th Avenue, New Yo 20). *Perfum. Essent. Oil Rec.*, 1960, **51** (10), 535 540.—The integration of spectrophotometric, i. and gas-chromatographic data together wit pharmacopoeial tests is discussed as a means detecting additions to natural bergamot oi Natural bergamot oil displays u.v. absorptio maxima at 270 mμ and 312·5 mμ ($E^{1\%}_{1cm}$ = 10 t 13); the extinction values are directly proportiona to the concn. of bergaptene, so that a low valu indicates dilution. In the i.r. spectrum, linal acetate absorbs at 12·52 μ and 12·00 μ; the norma ratio of the absorption at 12·00 μ to that at 12·52 is 0·9 to 1·14, a lowering of this ratio indicatin added terpenes. Chromatograms of the natura oil are characteristic and show the following are percentages—α-pinene, 7 to 10; (+)-limonene, 3 to 46; linalol, 17 to 24; and linalyl acetate, 22 t 30. H. B. HEATH

1652. **Analysis of civet. Investigation of cive with the aid of urea.** F. Wolf (Anal. Lab. VE Chem. Fabr. Miltitz, Leipzig). *Riechstoffe Aromen*, 1960, **10** (8), 261–266.—The sample dissolved in $CHCl_3$ and treated with a methanoli soln. of urea (*Ibid.*, 1960, **10**, 33; 69) to remov hydrocarbons, waxes, etc., as adducts, and th liquid residue is examined for aroma-impartin

ingredients. Procedures are given for the formation of the adduct and its isolation and decomposition with hot water, and for the separation of the liquid residue from the adduct and the removal therefrom of $CHCl_3$ and methanol. The original sample is analysed for matter insoluble in $CHCl_3$ at 50° and for matter volatile at 105°; the product obtained on decomposition of the adduct is analysed for yield on the original sample, m.p., iodine value, and acid and ester values; the liquid residue is analysed for yield on the original sample, refractive index, acid, ester and hydroxyl values and content of carbonyl compounds (determined by the hydroxylammonium chloride method and calculated as civetone). H. L. WHITEHEAD

1653. **Detection and analysis of chlorosulphonated polyethylene.** G. G. Esposito (Aberdeen Proving Ground, Md., U.S.A.). *Off. Dig. Fed. Paint Varn. Prod. Cl.*, 1960, **32**, 67–70.—A specific test for chlorosulphonated polyethylene, dependent on the reaction of SO_2Cl groups in the polymer, consists in adding to a solution of the resin (10 mg) in pyridine (3 ml), 2-aminofluorene (25 mg); a reddish colour develops within 1 hr. in the presence of the polymer. Sulphur and Cl may be determined after fusion with K_2CO_3 - Na_2O_2. The non-volatile content of the system, consisting of resin solution and curing agent, may be determined by stirring thoroughly, weighing large samples (5 to 10 g) into flat containers and heating for 3 hr. at 110°. L. A. O'NEILL

1654. **Polarographic determination of styrene in polystyrene.** J. Paściak (Chemical Works, Oświęcim, Poland). *Chem. Anal., Warsaw*, 1960, **5** (3), 477–486.—After a brief review of available methods, a study of the polarographic method is described. Solutions of styrene and polystyrene (20% in benzene) were extracted with (*i*) 0·05M-tetrabutylammonium iodide (**I**) in 75% ethanol and (*ii*) 75% ethanol. The extract was separated by centrifuging and polarograms were run on aliquots of (*i*) as obtained and aliquots of (*ii*) after mixing with an equal volume of 0·1M-**I** in ethanol, both before and after removal of O with N; (*ii*) gave more reliable results, but only 0·1% of styrene could be determined, and the ratio of wave height to concentration was not linear for values $< 1·6\%$. The errors were $\simeq 4\%$ for the range 0·1 to 4·5%. The substitution of dimethylformamide for ethanol resulted in improved sensitivity and accuracy and simplified the procedure. Polarograms are run on 2 ml of 0·1M-**I** in dimethylformamide to which 0·5 ml of the sample soln. in benzene (2·5 or 5%) has been added and O removed by passing a stream of N for 5 min.; $E_{\frac{1}{2}} = -2·46$ *vs.* the S.C.E. The calibration curve is linear throughout the whole range studied, and 0·03% of styrene can be determined with an error of $\simeq 1·5\%$. P. BRYCH

1655. **Hand-made shellac.** British Standards Institution (2 Park Street, London, W.1). B.S. 3280:1960. 38 pp.—Five grades are specified, with methods for sampling, determination of matter insol. in hot alcohol, orpiment, rosin, volatile matter, matter sol. in H_2O, non-volatile matter sol. in cold alcohol, ash, grit, wax, acid value, iodine value, arsenic, lead and physical properties.

1656. **Seedlac.** British Standards Institution. (2 Park Street, Lonodn, W.1). B.S. 3279:1960. 16 pp.—Six grades are specified, with methods for sampling, and the determination of volatile matter, wax, matter insol. in hot alcohol, non-volatile matter sol. in cold alcohol and matter sol. in water.

1657. **Determination of beeswax in wax compositions from the total paraffins, saponification, ester and acid values.** G. Spengler and A. Weber (Tech. Hochschule, München). *Fette, Seif., Anstrichmitt.*, 1960, **62** (10), 918–923.—A method is described in which the beeswax content can either be read from graphs of total paraffins, saponification, ester and acid values, or can be calculated from these results. The max. error is $\pm 3\%$. The presence of a foreign acid, or of a mixture of foreign acids or esters, can also be detected. I. DICKINSON

See also Abstracts—**1340**, Progress in elementary micro-analysis. Progress in functional-group micro-analysis. **1407**, Sulphate in detergents. **1425**, Decaborane in xylene. **1437**, Yttrium in coal ash. **1447**, Germanium and Ga in coal. **1473**, Ammonia in presence of amines. **1512**, Uranium in coal ash. **1530**, Detection of iodine in org. compounds. **1566**, Arsenic in coal and coke. **1760**, Dichlorophen on textiles.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

1658. **Tritium radioactivity determination of biological materials by a rapid dry-combustion technique.** E. A. Peets, J. R. Florini and D. A. Buyske (Pharm. Res. Dept., Expt. Therapeutics Res., Lederle Lab. Div., American Cyanamid Co., Pearl River, N.Y., U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1465–1468.—A furnace is described in which three samples of up to 2 g each can be simultaneously burned in O. The section of the furnace containing the catalyst (a roll of nickel foil and a pad of silver wool) is kept at 930° to 970° and the section containing the dried sample is heated steadily from room temp. to 700° in 30 min. Each of the three combustion tubes is connected to a cold trap. The tritium content of the collected water is determined in a liquid scintillation counter. The combustion of three samples takes 45 min. G. BURGER

1659. **Methods for the destruction of organic matter.** The Metallic Impurities in Organic Matter Sub-Committee of The Analytical Methods Committee of The Society for Analytical Chemistry. *Analyst*, 1960, **85**, 643–656.—The methods described are—(*a*) destruction with HNO_3 and H_2SO_4 with or without $HClO_4$ or H_2O_2; (*b*) destruction with HNO_3 and $HClO_4$; (*c*) destruction with HNO_3 and NH_4NO_3; and (*d*) special methods of wet decomposition for the determination of Hg. In the six methods described for dry-ashing, the ashing aids used are, respectively, none, MgO, $Mg(NO_3)_2$ soln., anhyd. Na_2CO_3, Na_2CO_3 soln. and H_2SO_4. The choice of ashing aid is governed by the nature of the sample or by the nature of the metal under investigation. In both wet and dry procedures, indications are given of the circumstances in which each procedure may be used. A. O. JONES

1660. **Titrimetric tetraphenylborate determination of potassium and application to blood serum and plant material.** F. Bermejo Martínez and Monserrat Gonalons Bori (Fac. of Sci., Univ.,

Santiago de Compostela, Spain). *Chemist Analyst*, 1960, **49** (3), 69–71.—To determine K in blood serum the sample is deproteinised with sodium tungstate and H_2SO_4. Plant material is digested with HNO_3 and H_2SO_4. *Procedure*—To 1 ml of soln. (0·006 to 3 mg of K) add 20% NaOH soln. to pH 9, 0·5 ml of 35% formaldehyde soln. and approx. 1 ml in excess of purified 1·2% Na tetraphenylborate (**I**) soln. in dil. NaOH soln. (pH 9). Dilute to 25 ml, set aside for approx. 3 min. and filter. Titrate 5 ml of the clear filtrate with a quaternary ammonium salt soln., with bromophenol blue as indicator. To standardise the soln. of **I**, mix 2 ml of KCl soln. [1·25 g of KCl, 50 ml of 4% ammonium oxalate soln. and 5 ml of aq. NH_3 (1:1), diluted to 250 ml] with 1 drop of 20% NaOH soln., 0·5 ml of formaldehyde soln. and 5 ml of **I** soln. Dilute to 25 ml and proceed as described above. P. D. PARR-RICHARD

1661. **Complexometric determination of potassium in the presence of ammonium.** A. Holasek, H. Lieb and M. Pećar (Univ. Graz, Austria). *Mikrochim. Acta*, 1960, (5–6), 750–754 (in German).—For the determination of K in urine by pptn. with $Na_3Co(NO_2)_6$ and complexometry of the Co in the ppt., the concn. of NH_4^+ must be $< 0·02$N; the NH_4^+ can then be masked with formaldehyde (**I**), 1 ml of 30% **I** soln. masking 720 μg of NH_4^+. The accuracy on 300 to 600 μg of K in 2 ml of 0·005N NH_4^+ soln. is within $\pm$ 1·6%. *Procedure*—To the sample (2 ml) add 50% Na acetate soln. (5 ml), **I**, and 30% $Na_3Co(NO_2)_6$ soln. (1 ml). Set aside overnight in the cold, filter and wash the ppt. with cold water. Dissolve the ppt. in boiling 0·25N-HCl (5 ml) containing a little urea, add 50% Na acetate soln. (0·5 ml) and heat to boiling. To the hot soln. add a few crystals of ascorbic acid, 2 drops of 0·1M-Cu - EDTA and 2 to 4 drops of 0·05% ethanolic 1-(2-pyridylazo)-2-naphthol soln. Titrate with 0·001M-EDTA (disodium salt) to a yellow end-point. J. P. STERN

1662. **Determination of magnesium in plant material with Titan yellow.** E. G. Bradfield (Res. Sta., Long Ashton, Bristol, England). *Analyst*, 1960, **85**, 666–670.—Since Titan yellow varies in composition, a spectrophotometric standardisation of its soln. in 0·1% aq. poly(vinyl alcohol) is made at 550 mμ to ensure adequate concn. of the component that precipitates Mg. The ground and dried plant material (1 g) is digested with HNO_3 and, after addition of $HClO_4$, heating is continued until nearly all the $HClO_4$ has been removed. The cooled residue is diluted, boiled and filtered, and the vol. is adjusted to 100 ml. An aliquot is adjusted to pH 3 to 4 with aq. NH_3 and applied to an exchange column of De-Acidite E (Cl^- form), and the column is eluted with water until 100 ml has been collected. An aliquot of the eluate and a series of standards (0 to 100 μg of Mg) are treated with 2 ml of a compensating soln. containing $CaCl_2$, $AlCl_3$, hydroxyammonium chloride and triethanolamine, and are heated in water at 25°, after which a 4-ml portion of Titan yellow - poly(vinyl alcohol) reagent is added and then immediately 4 ml of NaOH soln. (8% w/v). After 30 min. the extinctions are measured at 550 mμ. Results with some plant materials are quoted and compared with those found by the 8-hydroxyquinoline method. A. O. JONES

1663. **Acid Alizarin black SN, a metallochromic indicator for calcium.** R. A. Close and T. S. West (Univ., Birmingham, England). *Anal. Chim. Acta*, 1960, **23** (3), 261–267 (in English).—Acid Alizarin black SN (C.I. Mordant Black 25) (**I**) and Acid Alizarin black SE (C.I. Mordant Black 10) (**II**) have been examined as complexometric indicators. Only **I** is of any value and is stated to be the best indicator for Ca in dilute ($\gtrsim 0·02$M) soln. The colour change is from purple to red just before the end-point, which is marked by a very sharp change to blue. The preferred pH is 11·5 to 12·5; Sr behaves similarly, and Cd gives a sharp change from red to blue at pH 8·5. The change with Mn is from purple to blue at pH 10, and with Ni and Zn it is from purple to blue at pH 11·5 in hot soln. Thorium gives a sharp change from crimson to orange at pH 4. It is concluded from spectrophotometric evidence that the complex with Ca is a 2:1 chelate of indicator with Ca. H. M.

1664. **Photometric titration of calcium in blood serum with Acid Alizarin black SN as metallochromic indicator.** R. A. Close and T. S. West (Univ. Birmingham, England). *Anal. Chim. Acta*, 1960, **23** (4), 370–374 (in English).—The sample of serum (100 μl) is diluted to 2·5 ml, 0·2M-triethanolamine (1 drop), N-NaOH (0·5 ml) and 0·1% Acid Alizarin black SN (C.I. Mordant Black 25) soln. (3 drops) are added, and the soln. is titrated with $2·5 \times 10^{-3}$M-EDTA (disodium salt) in an E.E.L. photo-electric titrator with a filter at 660 mμ. T. R. ANDREW

1665. **Colorimetric determination of iron in plasma.** E. Scala, A. Castaldo and G. Ruiz (Ist. Chim. Biol., Univ. Napoli, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1960, **36** (19), 1048–1052.—The method involves liberation of iron from its globulin complex by treatment with hot dil. HCl, removal of protein by treatment with trichloroacetic acid, and colorimetric determination of Fe in the resulting soln. after addition of KSCN. It is preferable (but not essential) to use plasma treated with heparin as an anticoagulant. *Procedure*—Prepare a 0·5% soln. of heparin and transfer 0·2 ml to a centrifuge tube and dry in an incubator for 24 hr. To the residue add venous blood (4 ml), centrifuge immediately, and separate the plasma. To the plasma (2 ml) add 0·3N-HCl (1 ml) and place in a water bath at 56° for 20 min. Add 20% trichloroacetic acid soln. (2 ml), mix and centrifuge for 10 min. at 2000 r.p.m. Remove the clear supernatant liquid (2 ml) and add 29% KSCN soln. (0·8 ml) and a fresh satd. soln. of $K_2S_2O_8$ (0·2 ml). Measure the extinction at 390 mμ and compare with standards treated similarly. Interference by iron in haemoglobin is small ($< 0·02\%$ of the total Fe present) and the recovery of added Fe exceeds 95%. E. C. APLING

1666. **Macro- and micro-determination of traces of cobalt. VI. Determination of cobalt in blood.** W. Haerdi, J. Vogel, D. Monnier and P. E. Wenger (Lab. de Chimie Minérale, Univ. Genève). *Helv. Chim. Acta*, 1960, **43** (3), 869–877 (in French).—The Co in the sample is recovered as the nitroso-R salt complex and determined spectrophotometrically. Losses (which amount to 10 to 30% of the metal) are allowed for by addition of a known amount of ^{60}Co as an internal tracer. To 50 to 100 mg of blood is added 4 ml of a soln. of a ^{60}Co salt containing 0·003 μg of ^{60}Co (0·017 μC) and the mixture is evaporated in a quartz vessel under an infra-red lamp at 160° to 180°; the residue is heated in an oven at 450° to 500° to remove the carbon. The residue is dissolved by warming in 40 ml of 9M-HCl. The soln. is passed through a

owex 1-X8 column (10 g, washed, demineralised ith 0·01M-HCl and then saturated with 9M-HCl), e column is washed with 9M-HCl and the Co uted with 30 ml of 4M-HCl in 4-ml portions, of hich the second and third are taken for analysis; ese are evaporated in a quartz vessel, in the resence of 5% NaCl soln. under an infra-red lamp, d the organic residue is destroyed by heating ith 0·5 ml of HNO_3 (1·5 ml) and subsequently ith $HClO_4$. The residue is dissolved in 1 ml of Cl, again evaporated and dissolved in 0·4 ml of ater and 0·4 ml of 10% Na acetate soln. The radiation of the soln. is measured. This permits ie calculation of the actual Co loss. Finally, 1 ml of a 0·05% soln. of nitroso-R salt is added, llowed by 0·1 ml of HNO_3 and heating to destroy ie excess of reagent. The mixture is centrifuged : 3000 r.p.m. for 20 min. The extinction of the ln. is measured and the Co content calculated.

J. L. Prosser

1667. **Determination of vanadium and cobalt in lants by neutron activation analysis of paper hromatograms.** H. W. Yurow. *Dissert. Abstr.*, 960, **21** (1), 46–47.—A soln. of the plant ash at H 1·5 was treated with cupferron and $CHCl_3$ to xtract Cu, Fe, Ti, V and Mo. A further extraction at pH 4·0 with diethyldithiocarbamic acid (**I**) nd CCl_4 removed Co, Ni and Zn. Paper chromaography of the cupferron extract with ammoniacal utanol - dimethylglyoxime separated V from ther constituents. Chromatography of the **I** xtract with 1,2-dimethoxyethane - H_2O separated o from Zn and partially from Ni (which does not nterfere). The chromatograms were irradiated vith thermal neutrons which formed ^{52}V and ^{60}Co. The radioactivities of these isotopes ere measured with a Geiger - Müller counter and elated to the amount of element present. N. E.

1668. **Rapid estimation of uranium in urine sing the type 1080A fluorimeter.** U.K.A.E.A. Production Group, Chemical Services Dept., pringfields, Lancs., England). U.K.A.E.A. Report PG 57(S), 1960. 9 pp.—Full experimental details re given of a procedure in which the urine (1 ml) s evaporated to dryness and the residue, after gnition, is fused with a mixture of $NaHCO_3$ and NaF (9:1) at 1000° for 1 min. The cooled mass is hen examined in the fluorimeter and the content f U is derived by applying a factor that has been btained, either by calibration involving the use of nown quantities of U, or by making a standard ddition of U to two of the four samples analysed. The limit of detection is 5 μg of U per litre; the oefficients of variation for the two methods of alibration are 15 and 12%, respectively.

G. J. Hunter

1669. **Rapid ultra-violet spectrophotometric determination of salicylate in blood.** G. W. Stevenson Dept. of Pharmacol. and Toxicol., Sch. of Med., Univ. of Calif., Los Angeles, U.S.A.). *Anal Chem.*, 960, **32** (11), 1522–1525.—Salicylate (up to 50 mg per 100 ml) can be extracted from whole blood or ny blood fraction, and simultaneously acidified, by haking the sample with a soln. of malonic acid in utyl ether. The extinction of the extract is letermined at 378, 314, 307, 300 and 280 mμ, nd the salicylate concn. is calculated from given equations. A further extraction of the ether xtract with phosphate buffer is described, but offers no significant advantage. G. Burger

1670. **Comparison of radiochemical and colorimetric methods for the determination of sulphaphenazol in blood and tissues.** K. Schmid, J. Tripod and F. Gross (Forschungslab., CIBA, A.-G., Basel, Switzerland). *Klin. Wochschr.*, 1960, **38** (17), 862–866.—The colorimetric method of Bratton and Marshall (*J. Biol. Chem.*, 1939, **128**, 537) for sulphonamides, as applied to sulphaphenazol [5-(*p*-aminobenzenesulphonamido)-1-phenylpyrazole], is compared with a radio-isotope method with ^{35}S-labelled material. The methods agreed closely in the values obtained for concn. in blood, urine and faeces, but the radiochemical method gave very much higher results than the colorimetric method for tissues. The reason for this is undetermined. H. F. W. Kirkpatrick

1671. **Detection and determination of aniline in forensic studies.** E. A. Gryaznova and V. G. Belikov. *Sudebnomed. Ekspertiza*, 1959, **2** (4), 39–43; *Ref. Zhur., Khim.*, 1960, (12), Abstr. No. 47,256.—A new specific colour reaction is described for the detection and determination of aniline (**I**) in biological material with a soln. of sodium nitroprusside (**II**) irradiated with u.v. light. Separate the **I** from the biological material by steam-distillation; to 1 ml of the distillate add a few drops of 2% Na_2CO_3 soln. and 1 ml of 1% **II** soln. that has been irradiated with a quartz lamp for 20 min. At a concn. of **I** of 50 μg per ml a blue colour appears after a few seconds; at lower concn. the colour develops more slowly. There is no interference from HCN, $CHCl_3$, chloral, hexachlorocyclohexane, phenol, nitrobenzene, benzene, toluene or formaldehyde. To determine **I**, to 100 g of cadaveric material add a known amount of **I** (100, 150, 200, 250 or 300 μg) and, after setting aside for 2 or 3 hr., steam-distil; measure the vol. of distillate and to 10 ml add 3 ml of 2% Na_2CO_3 soln. and 7 ml of **II** soln.; after 20 min. measure the extinction in a Duboscq colorimeter, with a standard scale for comparison. The recovery of the **I** present in biological material is 73·5 to 93·5%, and the method is as sensitive as the bromatometric method. C. D. Kopkin

1672. **Detection and estimation of trace amounts of 3,4-benzopyrene in biological material.** J. Pavlů and J. Šula (Fac. of Med., Charles' Univ., Prague, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (9), 2461–2463 (in English).—The sample is treated with ethanolic KOH, set aside for 12 hr. and then heated under reflux for 5 hr. and then extracted with light petroleum. The extracts are washed with H_2O, dried over $CaCl_2$ and evaporated to dryness. The residue is dissolved in benzene and the soln. is chromatographed on a column of alumina, with benzene as the eluent. The eluate is concentrated and resolved by chromatography on acetylated paper with methanol - ether - water (4:4:1). The chromatograms are examined under u.v. light, the presence of 3,4-benzopyrene being shown by blue - violet spots. When the concn. is > 2 μg per 50 g of sample, the benzopyrene is determined absorptiometrically at 389 mμ on the eluate from the spots. The method is applicable to liver tissue and, with some modification, to anthracotic lymphatic nodes. G. P. Cook

1673. **Diagnostic composition [for oral administration to determine gastric acidity].** Cilag-Chemie, Ltd., formerly Cilag, Ltd. Brit. Pat. 850,426; date appl. 28.10,58. Switzerland, date appl. 5.11.57.—The composition comprises 2,6-diamino-3-phenylazopyridine, or a 3-(*p*-alkylphenylazo)- or

a 3-(*p*-alkoxyphenylazo)-2,6-diaminopyridine in which the alkyl or alkoxy-groups contain 1 to 4 carbon atoms, in admixture with an amphoteric protein (zein) that is practically insol. in water and in N-HCl and of which the iso-electric point lies at a pH below 7. The amount of dye excreted is dependent on the pH conditions in the stomach, and is determined colorimetrically on a sample of urine, 1·5 hr. after ingestion, after acidification with HCl. J. M. Jacobs

1674. **Colorimetric determination of 3,6-anhydrogalactose and galactose in marine algal polysaccharides.** W. Yaphe (Atlantic Reg. Lab., National Res. Council, Halifax, Nova Scotia, Canada). *Anal. Chem.*, 1960, **32** (10), 1327–1330.—To determine 3,6-anhydrogalactose (**I**), mix the sample soln. (2 ml, containing 5 to 40 μg of **I** or 30 to 200 μg of polysaccharides) with reagent [add 0·13% ethanolic resorcinol soln. (10 ml) to 12M-HCl (100 ml) immediately before use] (10 ml), mix, heat for 10 min. at 80° ± 0·2°, cool in ice for 90 sec. and measure the extinction at 550 mμ. To determine **I** and galactose (**II**) together, and hence **II** by difference, cool a 0·2% soln. of anthrone in 80% (v/v) H_2SO_4 (10 ml) for 5 min. in ice, add the sample soln. (2 ml), set aside in ice for 2 min., mix well, heat at 80° for 20 min., cool in ice for 5 min. and measure the extinction at 625 mμ against a reagent blank. Alternatively, use the extinction of soln. heated with anthrone reagent for 5 min. and 20 min. to calculate the concn. of **I** and **II** simultaneously by the equations of Brown *et al.* (*J. Lab. Clin. Med.*, 1957, **49**, 630). The accuracy by either method is within $\simeq \pm 6\%$. A. R. Rogers

1675. **Thiamine and its mono-, di- and triphosphoric ester content of normal rat tissues.** G. Rindi and L. de Guiseppe (Ist. Fisiol., Univ. Pavia, Italy). *Experientia*, 1960, **16** (10), 447 (in English).—A protein-free extract of tissue homogenised in 5% trichloroacetic acid is adjusted to pH 6·7 to 6·8 with 40% NaOH soln. and passed through a charcoal column. After being washed with H_2O, the column is eluted with 60 to 70 ml of 10% n-propanol, and the eluate is concentrated to 5 ml at 25° to 30° *in vacuo*. The concentrate and washings (15 ml), after addition of 0·8 ml of 0·1N-HCl, are chromatographed on a column of Dowex 1-X8 (acetate form), which is eluted with 10 ml of H_2O. The thiamine and its monophosphate are determined in this eluate by difference before and after Taka-diastase digestion. The thiamine pyrophosphate is eluted from the column with 20 ml of 0·02M-Na acetate in 0·04M-acetic acid, and the triphosphate with 20 ml of M-acetate buffer at pH 4·5. After Taka-diastase hydrolysis, the pyrophosphate content is determined directly, and the triphosphate content after percolation through Amberlite IRC-50 buffered at pH 4·5. Recovery of the extracted thiamine compounds is about 95%. D. E. Evans

1676. **Two new solvent systems for the countercurrent distribution of carotenoids.** A. L. Curl (Western Utilisation Res. and Devel. Div., U.S. Dept. of Agric., Albany, Calif.). *J. Agric. Food Chem.*, 1960, **8** (5), 356–358.—A clean separation of diol, monoepoxide diol, diepoxide diol and polyol fractions was obtained with light petroleum - methanol (26·5 : 73·5) by countercurrent distribution in a Craig apparatus (*cf. Ibid.*, 1953, **1**, 456). The carotenoid polyols can be further fractionated by light petroleum - acetone - methanol - water (1·25 : 1·0 : 0·10 : 0·65). Graphs are given showing the distribution of the diols and polyols contained in orange juice and pulp, cling peaches, and pyracantha leaves. Spectral absorption maxima in benzene and N_{100} values (N_{100} = tube no. of max. per 100 transfers) are also given for individual fractions. R. A. Hendey

1677. **Determination of urinary porphyrins.** H. Holland (Katharinenhospital-Apotheke, Stuttgart, Germany). *Dtsch. ApothZtg*, 1960, **100** (38), 1096–1098.—A review is presented of methods for the rapid routine paper-chromatographic separation of the methyl esters of uroporphyrins I and III, coproporhyrins I and III, and protoporphyrin IX, and for the determination of the porphyrins and porphobilinogen in urine. (17 references.) A. R. Rogers

1678. **Infra-red absorption analysis of tissue constituents, particularly tissue lipids.** H. P. Schwarz. *Adv. Clinical Chem.*, 1960, **3**, 1–34.—A review article.

1679. **Chromatography of lipids on silicic acid.** J. J. Wren (Lyons Laboratories, Hammersmith Road, London, W.14). *J. Chromatography*, 1960, **4** (3), 173–195.—A review is presented with 290 references. The items dealt with comprise the adsorbent, the eluents, experimental considerations and the order of elution of the different lipid classes. R. M. S. Hall

1680. **Improved method for the detection of lipid on paper chromatograms.** L. L. Sulya and R. R. Smith (Sch. Med., Univ. Mississippi, Jackson, Tenn., U.S.A.). *Biochem. Biophys. Res. Commun.*, 1960, **2**, 59–62.—When paper chromatograms of lipids are dipped in a soln. of protoporphyrin in HCl and, after being washed in water, are examined in u.v. light, the lipids appear as brilliant red spots on a blue background. The wide range of lipids detectable by this technique includes fatty acids, triglycerides, lecithin, lysolecithin, sphingomyelin, cholesterol, and cholesteryl esters. Nutr. Abstr. Rev.

1681. **Micro-determination of long-chain fatty acids in plasma and tissues.** V. P. Dole and H. Meinertz (Rockefeller Inst., New York 21, U.S.A.). *J. Biol. Chem.*, 1960, **235** (9), 2595–2599.—Modifications of the original method of Dole (*J. Clin. Invest.*, 1956, **35**, 150) are given. A single extraction of plasma with the two-phase system heptane - isopropyl alcohol - water has sufficient analytical specificity for the subsequent determination of long-chain fatty acids. If exceptional amounts of lactic, acetic, acetoacetic or β-hydroxybutyric acids are present they are eliminated by a second extraction. The two-phase system is also useful for countercurrent distribution. A table of partition coeff. for various acids is given from which the number of transfers needed for analytical work can be calculated. A simple eight-transfer procedure, which needs < 0·5 hr., separates the acids of an extract into long-chain fatty acids, cephalins and polar org. acids. J. N. Ashley

1682. **Detection of some urinary amines.** J. Ratcliffe and P. Smith (R.A.F. Inst. of Aviation Med., Farnborough, Hants, England). *Chem. & Ind.*, 1960, (37), 1159.—4-Methoxyadrenaline, 4-methoxynoradrenaline and monohydroxy- and 4-hydroxy-3-methoxyphenyl-ethylamines, and, provisionally, 4-hydroxyphenylethanolamine (octop-

ne) and phenylephrine, its *N*-methyl homo- ie, have been detected in urine. The amines t 15-min. excretion aliquot were gently hydro- d from the sulphate forms and then adsorbed an ion-exchange resin (Na^+ form). After elution n 3N-aq. NH_3 the amines were separated from many non-basic substances and identified by er chromatography. R. M. S. Hall

683. **Water-elution chromatography of amino- ds on ion-exchange materials.** D. L. Buchanan . R. T. Markiw (Veterans Admin. Hospital, st Haven, Conn., U.S.A.). *Anal. Chem.*, 1960, (11), 1400–1407.—Several ion-exchange resins weak or intermediate acidity or basicity have n examined for their ability to separate mixtures amino-acids with H_2O as the eluent. Several nplete and many incomplete separations are orted. The results indicate the possibility of ieving many more complete separations by the of more suitable resins or blends of resins. G. Burger

684. **Determination of carbon and its radio- ivity. III. Transfer of small samples of carbon xide to counting tubes.** D. D. Van Slyke and Plazin (Med. Res. Centre, Brookhaven Nat. ., Upton, N.Y., U.S.A.). *J. Biol. Chem.*, 1960, (9), 2749–2752.—A micro-manometric modifi- ion of the ninhydrin - CO_2 method for the ermination of the carboxyl CO_2 of α-amino-acids described, together with a procedure for the nsfer of the CO_2 to a Bernstein - Ballentine counter for counting the $^{14}CO_2$. The method applicable to the determination of 10 to 30 μg carboxyl C. When CO_2 is transferred from a nometric chamber to a gas counter as described Van Slyke *et al.* (*Brit. Abstr. C*, 1952, 82), a all vol. of CO_2, sufficient to be significant with mples of < 2 mg of C, may be trapped in the nnecting tube. This can be washed quant. into counter by a few ml of methane, admitted into manometric chamber after the usual transfer eration. J. N. Ashley

685. **Ninhydrin as a reagent for *N*-methylamino- ds.** D. W. Russell (Plant Chem. Div., D.S.I.R., lmerston North, New Zealand). *J. Chromato- phy*, 1960, **4** (3), 251–252.—*N*-Methyl deriva- es of glycine, valine, leucine and isoleucine have en tested. Peart's method (*Biochem. J.*, 1956, 520) has been used for quant. determination. r qual. use a 1:1-mixture of 0·33% ninhydrin in utyl alcohol and glacial acetic acid - water - ridine (1:5:5) has been used and the paper is ated to 110° for 15 min. The limit of detection 0·01 μmole. Both amino- and *N*-methylamino- ids give purple colours; *N*-methylamino-acids are stinguished by the very faint colours given with nhydrin in water-satd. n-butanol containing 2% glacial acetic acid. R. M. S. Hall

686. **Paper-chromatographic identification of clic imino-acids.** G. Parmentier and H. Vander- eghe (Rega Inst., Univ., Louvain, Belgium). *Chromatography*, 1960, **4** (3), 228–232.—The sitions on a chromatogram are illustrated and e R_F values are tabulated for these compounds ter two-dimensional development with (*i*) n- tanol - acetic acid - water (4:1:5) and (*ii*) water- td. phenol. This order yields better results than e reverse one. Confirmatory colour reactions th various reagents, *viz*, ninhydrin, isatin, nhydrin - isatin - triethylamine (Kolor and berts, *Anal. Abstr.*, 1958, **5**, 652) and *p*-nitro- benzoyl chloride - pyridine (Plattner and Nager, *Helv. Chim. Acta*, 1948, **31**, 2203) are also tabulated and discussed. R. M. S. Hall

1687. **Qualitative separation of choline esters by means of high-voltage paper electrophoresis.** E. Heilbronn and B. Carlsson (Res. Inst. of National Defence, Sundbyberg, Sweden). *J. Chromatography*, 1960, **4** (3), 257–259.—The mobilities are reported of 18 choline esters in buffer soln. of pH 10·0, 9·1, 5·0 and 4·6 for electrophoresis at 12° to 13° for 30 to 60 min., at 30 V per cm (1500 V). After drying and spraying with dipicrylamine, choline and acetylcholine gave blue - red spots, and other choline esters gave orange - red ones. Molybdophosphoric acid is more reliable, but less sensitive, than dipicrylamine. R. M. S. Hall

1688. **Improvements in test compositions for protein [in biological fluids].** A. Galat. Brit. Pat. 852,669; date appl. 5.11.58.—A reagent for the detection of albumin in urine comprises a solution of an ester (ethyl ester) of bromophenol blue (in the form of a salt, *e.g.*, the potassium salt) and a weak org. acid (acetic acid) in a water-sol. tertiary alcohol (t-butyl alcohol, or diacetone alcohol). J. M. Jacobs

1689. **Albumin diagnostic compositions.** Miles Laboratories, Inc. Brit. Pat. 853,643; date appl. 20.3.59. U.S.A., date appl. 25.3.58.—An indicator for determining albumin in urine, etc., comprises strips of filter-paper impregnated with a soln. consisting of 10 ml of a buffer, composed of 2M-sodium citrate (2·2 pt.) and 2M-citric acid (7·8 pt.), and 10 ml of a 0·08% soln. of bromophenol blue in 95% ethanol. (Addn. to Brit. Pat. 826,066; *cf. Anal. Abstr.*, 1960, **7**, 1871.) J. M. Jacobs

1690. **Optimal staining conditions for the quantitative analysis of human serum protein fractions by cellulose acetate electrophoresis.** C. J. Brackenridge (Royal Perth Hosp., W. Australia). *Anal. Chem.*, 1960, **32** (10), 1353–1356.—The use of Lissamine Green (C.I. Acid Green 5) with the electrophoretic method of Kohn (*Clin. Chim. Acta*, 1958, **3**, 450) is investigated and its suitability for quantitative work is established. The dye-binding capacities of five protein fractions are determined, and are virtually linear with respect to concn. The recommended pH for the dye bath is 2·7, and for the eluting soln. 6·0. Temp. has a marked effect on the dye uptake and thermostatic control is essential to the staining process. H. F. W. Kirkpatrick

1691. **Optimal fractionation conditions for the quantitative analysis of human serum protein fractions by cellulose acetate electrophoresis.** C. J. Brackenridge (Royal Perth Hosp., W. Australia). *Anal. Chem.*, 1960, **32** (10), 1357–1359.—The variables investigated are the choice of buffer, its ionic strength and pH and the vol. used; the effects of voltage and running time of electrophoresis, of the length of strip, and of the position of application of the serum. The final technique is described in detail. H. F. W. Kirkpatrick

1692. **Comparative studies on protein hydrolysis with a view to reducing the destruction of components of the protein. I. Quantitative estimation of amino-acids in protein hydrolysates with the aid of an automatic recording apparatus.** G. Krampitz (Inst. Anat. Physiol. Haustiere, Univ. Bonn). *Z. Tierernähr.*, 1960, **15**, 40–46.—The apparatus is described.

II. Behaviour of free amino-acids in conditions of protein hydrolysis. G. Krampitz. *Ibid.*, 1960, **15**, 76–86.—Of the amino-acids studied, only tryptophan, cysteine and methionine were detectably degraded by treatment with 6N-HCl for 24 hr. under the conditions for protein hydrolysis.

III. Hydrolysis of pure proteins. G. Krampitz. *Ibid.*, 1960, **15**, 87–95.—A combination of conditions of hydrolysis was devised for bovine serum albumin to give complete recovery of all amino-acids. NUTR. ABSTR. REV.

1693. **Cystine content of proteins, foods and feeds. Comparison of chromatography on a sulphonated polystyrene resin and microbiological methods of determination.** R. J. Evans, S. L. Bandemer and D. H. Bauer (Dept. of Agric. Chem., Michigan State Univ., East Lansing, U.S.A.). *J. Agric. Food Chem.*, 1960, **8** (5), 383–386.—The cystine contents of several proteins, foods and feeding-stuffs have been determined by chromatography (*cf.* Schram *et al.*, *Anal. Abstr.*, 1954, **1**, 1936) and three microbiological procedures. In the microbiological procedures, *Leuconostoc mesenteroides* P-60 was used as the organism in the peptone medium method (Lyman *et al.*, *Arch. Biochem.*, 1946, **10** 427), with a 30-min. and a 6-hr. hydrolysis period, and, in the lanthionised casein method (*cf.* Horn and Blum, *Cereal Chem.*, 1956, **33**, 18), with a 30-min. hydrolysis time. Chromatography of cysteic acid gave the most reproducible results, and was considered the most satisfactory. The two microbiological procedures with a 30-min. hydrolysis period generally gave higher results than did chromatography, while the 6-hr. hydrolysis method gave lower results. Reasons are suggested for the erratic results of the microbiological assays, and tables of results are given for the cystine content of many foods and feeding-stuffs by using all the methods described.
R. A. HENDEY

1694. **Variable dye uptake in the quantitative analysis of abnormal globulins by cellulose acetate electrophoresis.** C. J. Brackenridge (Royal Perth Hosp., W. Australia). *Anal. Chem.*, 1960, **32** (10), 1359–1360.—The concn. of an abnormal protein is best determined by an independent method, as the dye uptake appears to be impossible to predict.
H. F. W. KIRKPATRICK

1695. **New paper-chromatographic technique for the separation of cholesterol and cholesteryl esters.** Č. Michalec and J. Strašek (Lab. of Protein Metab. & Proteosynthesis, Charles' Univ., Prague, Czechoslovakia). *J. Chromatography*, 1960, **4** (3), 254–257.—The reversed-phase partition system has been further modified. The paper (Whatman No. 3) is drawn through a soln. of 0·5% (v/v) of liquid paraffin (sp. gr. 0·880) in ether. After a few minutes the strips are ready and can be kept for more than 1 month. Development with acetic acid satd. with liquid paraffin for 5 to 6 hr. is used for C_2 to C_8 derivatives, and with acetic acid - $CHCl_3$ - liquid paraffin (16:1:1) for 3 to 4 hr. for cholesterol derivatives. By this procedure, the mobilities of the compounds differ more, and complete resolution is attained more rapidly; no previous equilibration is necessary. Saturated and unsaturated compounds cannot be resolved with these systems alone.
R. M. S. HALL

1696. **Characterisation of some steroid hormones through micro-sublimation.** E. Kassau (Univ.-Klinik., Bonn, Germany). *Dtsch. ApothZtg*, 1960, **100** (38), 1102–1106.—The appearance of the sublimates is described and limits of detection (mostly $< 1\ \mu g$) are given. There are 45 photomicrograp
A. R. ROGERS

1697. **Determination and significance of natural oestrogens.** J. B. Brown. *Adv. Clini Chem.*, 1960, **2**, 157–233.—A review article.

1698. **Effect of interfering chromogens in spect photometric determination of ruminant urina androgens.** W. S. Ruliffson, A. M. Gawienow and J. S. Hughes (Agric. Exp. Sta., Kansas Sta Univ., Manhattan, U.S.A.). *J. Agric. Food Che* 1960, **8** (5), 395–397.—The effect of certain nc steroid ketones and non-17-oxosteroids has be investigated with respect to the determination androsterone (**I**) and dehydroepiandrosterone (**I** Absorption spectra are given for the colours p duced in the Allan, Pincus and Zimmermann actions, with pure **I** and **II** and ruminant uri with added **I** and **II**. In each case the absorpti peaks, although characteristic, were different ar in general, moved to shorter wavelengths. U identified substances normally present in the uri are responsible for these effects and may enhance change the normal spectra of small amounts oxosteroids present in the urine.
R. A. HENDEY

1699. **Method for determination of 21-deoxyke steroids.** P. Cristol and M. F. Jayle (Fac. Médicine, 45 rue des Saints-Pères, Paris). *Bu Soc. Chim. Biol.*, 1960, **42** (5–6), 655–663.— method is discussed and developed whereby deoxyketol steroids (**I**) are reduced to the acetalc hydogenic steroids by means of alkali borohydric The acetaldehyde formed is determined by t method of Cox [*Biochem. J.*, 1952, **52**, 340 (macı method); *Anal. Abstr.*, 1960, **7**, 1879 (micı method)]. *Procedure with urine* (*macro-method*) Collect a 24-hr. sample of urine, with 1 ml of 2 $Hg(CN)_2$ soln. and 2 ml of $CHCl_3$ as antisepti To 25 ml add 2·5 ml of acetate buffer (pH 5·2) a 0·25 ml of *Helix pomatia* digestive juice (containi 10^5 Fischmann units of β-glucuronidase per ml, a a similar amount of sulphatase) and incubate f 24 hr. at 37°. Extract on a rotatory shaker for min. with 14 ml of $CHCl_3$ or dichloromethane, a twice more with 7 ml of $CHCl_3$ (15 min. each tim Wash the mixed $CHCl_3$ extracts 3 to 5 times wi 2·5 ml of 0·1N-NaOH (5 min. each time), then times with 2·5 ml of H_2O (4 min. each tim Evaporate the extracts to dryness, re-dissolve t residues in 5 ml of ethanol and divide the sol into two parts. In the first, determine the steroi having a propane-1,2-diol chain by the method Cox. To the second, add 0·2 ml of 5% aq. KB or $NaBH_4$ soln., set aside for 1 hr., then add 0·2 of acetic acid and set aside for 10 min. wi occasional shaking. Dry *in vacuo* on a boilin water bath, re-dissolve the residue in 0·2 ml acetic acid and determine the reduced steroid the method of Cox. The difference between t two results corresponds to **I**. The micro-meth may be used with one-tenth of the above quantitie Experiments with normal urine showed an avera recovery of 93·5%, with a range of 80 to 112% Results are quoted for normal and pathologic urines.
R. A. HENDEY

1700. **Assay of catalase for commercial use.** Scott and F. E. Hammer (Fermco Laboratorie Inc., 4941 S. Racine Av., Chicago, Ill., U.S.A *Enzymologia*, 1960, **22** (3), 194–200 (in English).— A comparison is made between the Keil meth (Armour & Co., Bulletin EC-2, 1954), based on t

mposition of H_2O_2 in an inert atmosphere, the Baker method (not previously described) vhich an aliquot of catalase soln. (≯ 1 ml aining ≯ 3·5 units) is allowed to act upon ml of H_2O_2 soln. (0·5M, buffered with phosphate H 7·0) at 25°, until the reaction is complete; of this soln. is titrated with 0·25N-$Na_2S_2O_3$ he presence of H_2SO_4, KI and ammonium rbdate. A blank is performed. One Baker is the amount of catalase required to decompose ng of H_2O_2 under the conditions of assay. The t of N or CO_2 as the inert gas, and of the aliquot is studied in the Keil method, which is shown e satisfactory for liver catalase but not for *rgillus* catalase. The effect of aliquot size on Baker method is considered, and this method own to be satisfactory for both liver and fungal lase. M. A. Ellis

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e also Abstracts—**1340**, Determination of ele- ts in biological systems. **1417**, Determination ysteine. **1771**, Partition of plant constituents.

Pharmaceutical analysis

701. **Quantitative isolation of alkaloids from t materials.** Kum-Tatt Lee (Dept. of Chem., . of the State of Singapore, Outram Road, apore). *Nature*, 1960, **188**, 65–66.—Isolate plant alkaloids as their insol. reineckates, olve the ppt. in acetone, and regenerate the loids by passage through a column of De- ite FF (OH^- form). Then determine the loids in the eluate by conventional procedures. method has been applied to the isolation of loids from opium and nux vomica, and to the rmination of papaverine and strychnine.
A. R. Rogers

702. **Assay of the sulphates of some alkaloids on-aqueous medium.** M. Rink, R. Lux and Riemhofer (Pharm. Inst., Univ., Bonn, many). *Dtsch. ApothZtg*, 1960, **100** (42), 1231– .—Dissolve the sample (≃ 1 milli-equiv.) in n acetic anhydride (10 ml), cool and titrate 0·1N-$HClO_4$ in H_2O-free acetic acid. Good lts are obtained with the sulphates of quinine, idine, sparteine, strychnine, codeine and pine. Brilliant green and Fettblau B (C.I. ent Blue 19) are generally suitable as indicators.
A. R. Rogers

703. **New colour reaction for the detection of ro quantities of strychnine.** N. Stamenova. *natsiya*, 1959, **9** (5), 19–24 (in Bulgarian); *Ref. ., Khim.*, 1960, (17), Abstr. No. 23,733.—In analysis of mixtures of atropine with strychnine the Vitali - Morin reaction in an acetone ium, atropine develops a violet coloration h, on the addition of water, is discharged and lden-yellow colour develops. Pure strychnine ted by the same method gives a similar final ration, which is specific to strychnine. Brucine s a similar colour, but only after a blood-red se which strychnine does not produce. The nod is used for the detection of strychnine he presence of morphine, atropine, hyoscine, ine, veratrine, ethylmorphine, cocaine and ine. The reaction is sensitive to 5 μg of chnine. A. Burwood-Smith

704. **Modified colorimetric determination of rpine in pure preparations and tablets.** W. owicz and N. Twierdochlebow (Chemical Works, Pabianice, Poland). *Chem. Anal., Warsaw*, 1960, **5** (3), 401–406.—Reserpine, both as solid and in $CHCl_3$ soln., reacts with bromophenol blue at pH 4 (phosphate - citrate buffer) to form a water-insol. yellow compound which is extracted with $CHCl_3$. The extract is then shaken with 0·1N-NaOH, and the liberated bromophenol blue in the aq. layer, diluted with an equal vol. of 95% ethanol, is measured in a photo-electric colorimeter with a green filter. The error is ± 1% on 0·015 mg of reserpine in 20 ml. P. Brych

1705. **Scintillation radio-autography of tritium-labelled compounds on paper chromatograms.** E. V. Parups, I. Hoffman and H. R. Jackson (Res. Branch, Canada Dept. of Agric., Ottawa). *Talanta*, 1960, **5** (2), 75–77 (in English).—Tritiated nicotine can be determined by dipping the chromatogram momentarily in a soln. of a scintillating compound (a satd. soln. of anthracene in benzene), allowing it to dry and placing it in contact with fast film (Kodak Royal-X Pan), which is exposed for 1 to 2 weeks. The spot obtained on the film is compared by means of a densitometer with spots obtained from known amounts of tritiated nicotine; 0·38 and 0·19 μC per sq. cm can be detected with exposures of 1 and 2 weeks, respectively.
W. T. Carter

1706. **Colorimetric determination of santonin in artemisia.** R. A. Khan and H. Mohiuddin [Glaxo Lab. (Pakistan) Ltd., Karachi]. *J. Pharm. Pharmacol.*, 1960, **12** (9), 544–551.—Santonin reacts with hydroxylamine and $FeCl_3$ to give a purple colour which obeys Beer's law over the range 1 to 5 mg per ml. The results for 5 samples of artemisia agree well with those obtained by the gravimetric method of Massagetov (*Arch. Pharm.*, 1932, **270**, 392) and are about 3% less than those obtained by a volumetric method based on that of Bohme (*Arch. Pharm.*, 1942, **280**, 89). Other lactones, such as artemisin, α-santonin and ψ-santonin give similar colours. *Procedure*—Extract a mixture of the finely powdered flowerheads (2 g) and CaO (0·5 g) with boiling water, extract the santonin with $CHCl_3$ and decolorise the extract by heating with animal charcoal (0·1 g). Evaporate to dryness, dissolve the residue in 18% ethanol, heat again with animal charcoal (0·1 g), filter and adjust the concn. of ethanol to 50% in a total vol. of 50 ml. Mix a 3-ml aliquot with a fresh 7·5% soln. of hydroxylamine in N-NaOH (2 ml), add N-NaOH (5 ml), set aside for 5 min., titrate with N-HCl, to a colourless end-point, with 2:4-dinitrophenol as indicator, and dilute to 50 ml with H_2O; mix a 5-ml aliquot with 2% $FeCl_3$ soln. (1 ml) and measure the extinction within 3 min. at 500 mμ.
A. R. Rogers

1707. **Determination of ascaridole in chenopodium oil with hydrogen bromide in acetic acid.** M. I. Blake and R. E. O'Neill (Chem. Div., Argonne Nat. Lab., Lemont, Ill., U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1370–1371.—*Procedure*—Shake the oil (150 to 200 mg) with glacial acetic acid (20 ml) and 0·1N-HBr in acetic acid (25 ml) for 5 min., set aside at room temp. for 24 hr. and titrate potentiometrically with 0·1N-Na acetate. Perform a blank determination. The results of 7 assays of one sample of oil ranged from 64·56 to 65·83%; the results of 7 assays of the same sample by the [U.S.] N.F. 1955 method ranged from 64·04 to 66·95%.
A. R. Rogers

1708. **The analysis of ternary mixtures by thermo-microscopic methods.** M. Brandstätter-Kuhnert and J. Obkircher (Pharm. Inst., Univ., Innsbruck, Austria). *Mikrochim. Acta*, 1960, (5–6), 836–853 (in German).—The ternary mixtures carbromal - phenacetin - salicylamide and phenazone - phenacetin - phenobarbitone were studied. By plotting the refractive index isotherms of the fused mass and the isotherms of the primary crystals on triangular concn. diagrams, the influence of each factor upon the quant. determination could be found. In each case there was an area unsuitable for measurement where the isotherms were practically parallel; when this was not the case, quant. determination was possible. Precautions should be taken to prevent loss of highly volatile components. P. D. PARR-RICHARD

1709. **Colorimetric method for the determination of amethocaine alone or in mixtures with atropine or procaine.** N. Stamenova. *Farmatsiya*, 1959, **9** (5), 16–19 (in Bulgarian); *Ref. Zhur., Khim.*, 1960, (17), Abstr. No. 23,732.—The Vitali - Morin colour reaction for atropine is modified specifically for amethocaine (**I**). In the analysis of mixtures of atropine and **I** in an acetone medium, atropine develops a characteristic violet coloration. On the addition of methanol the violet colour is discharged, and a stable ruby-red colour develops. In the analysis of pure **I** by the same method a similar final coloration is observed. It is established that this coloration is specifically due to **I**; pilocarpine, psicaine, cocaine, procaine, cinchocaine and benzocaine do not give a coloration. Beer's law is obeyed, and the reaction is sensitive down to 10 μg of **I**. It is possible to detect **I** in all combinations with procaine and to determine **I** quant. when the ratio of **I** to procaine is $> 1:1$.
A. BURWOOD-SMITH

1710. **Quantitative determination of sulphonamides with ion-exchange paper.** E. Lewandowski (Inst. f. allg. Chem., Univ. Poznań, Poland). *Anal. Chim. Acta*, 1960, **23** (4), 317–321 (in German).—Sulphonamides may be separated chromatographically on cation-exchange paper. The zones are rendered visible by spraying with *p*-dimethylaminobenzaldehyde and the area of each zone is directly related to the amount of material present, *e.g.*, for 50 to 200 μg of sulphanilamide, sulphaguanidine and sulphacetamide. T. R. ANDREW

1711. **Determination of Paludrine [proguanil].** P. Spacu and I. Albescu (Acad. R.P.R., Bucharest). *Acad. R.P.R., Stud. Cercet. Chim.*, 1960, **8** (1), 91–96.—The method is based on the production of a complex with Ni^{2+}. *Procedure*—Between 0·070 and 0·25 g of the sample is dissolved in 30% ethanol and treated with a slight excess of an ammoniacal soln. of $NiCl_2$. The ppt. is collected in a IG4 crucible, washed with cold water and dried at 110°, or dissolved in hot ethanol, and measured spectrophotometrically at 430 mμ. The gravimetric method gives errors of $\simeq \pm 3{\cdot}0\%$.
H. SHER

1712. **Determination of methadone and pethidine in some galenical preparations. I. Tablets.** T. Rutschmann (Lab. Pharm. Galénique, Univ., Genève). *Pharm. Acta Helv.*, 1960, **35** (9–10), 482–487 (in French).—*Procedure*—Extract the powdered tablets containing $\simeq$ 150 mg of methadone hydrochloride or $\simeq$ 300 mg of pethidine hydrochloride by shaking for 10 min. at 55° with a mixture of 10% HCl - H_2O (2:3) (30 ml) and filter hot. Wash the residue and filter with hot aq. HCl, cool the combined filtrate and washings and dilute 100 ml. Add aq. NaOH soln. to a 50-ml aliq until alkaline, extract with several portions ethyl ether, wash the combined extracts with and reject the washings. Evaporate the extr to dryness and titrate the residue with HCl. precision is 3 to 4% in the presence of sta stearates, alginates and carboxymethylcellul Low results may be obtained in the presence colloidal silicic acid or polyoxyethylene glycol.
A. R. ROGER

1713. **Determination of Synopen (halopyramin** P. Spacu and E. Antonescu (Acad. R.P.R., Buc rest). *Acad. R.P.R., Stud. Cercet. Chim.*, 196 (1), 73–83.—A colorimetric method based on the p duction of a complex from ammonium reineck (**I**) and halopyramine (**II**) is proposed. *Procedur* The acid extract of **II** is treated with a 1% aq. so of **I** in the cold. The ppt. is washed with 15 m water, then dissolved on the filter with acetone, a the extinction of the deep-red soln. is determined 533 mμ and compared with standards. The m error for the range 0·7 to 1 mg of **I** per ml is — 6·0 Gravimetric, spectrophotometric and potent metric methods are based on the use of the comp formed with $K_3[Cr(SCN)_6].4H_2O$ (**III**). For gra metric determinations, the slightly acid soln. **II** is treated with a conc. soln. of **III** in the c until the supernatant liquid is violet. Af shaking the mixture for 5 min., the ppt. is collect washed with water till free from Cl^- and dried 120°. The method has been used for determi tions on 0·3 mg with an error of + 1%. spectrophotometric determinations, the ppt. dissolved in acetone and the max. extinction measured with a 574-mμ filter. For the potent metric method, the ppt. is washed free from and dissolved in 20 ml of acetone. The soln. diluted with water to a slight opalescence a treated with a further 10 ml of acetone. titration is made with a silver indicator electr (in a soln. of 0·05N-$AgNO_3$) and a calomel refere electrode. H. SHE

1714. **Gravimetric micro-determination of Flaxe (gallamine triethiodide).** P. Spacu and E. Antones (Chem. Res. Centre, Acad. R.P.R., Buchare *Acad. R.P.R., Stud. Cercet. Chim.*, 1960, **8** (1), 1 180.—The method is based on the production an insol. complex from gallamine triethiodide a $K_3[Cr(SCN)_6]$ (**I**). *Procedure*—The sample is aci fied with 5 to 6 drops of conc. HCl, then treat with a conc. soln. of **I** until the soln. is violet. mixture is filtered after 5 min. and the ppt. is ov dried at 120°. The error is $\pm$ 4%. H. SHE

1715. **Polarographic determination of 4-ami 1,3-dimethyl-5-nitrosouracil.** H. Marciszew and A. Grzeszkiewicz (Pharm. Inst., Wars Poland). *Chem. Anal., Warsaw*, 1960, **5** (3), 5 510.—The dissolution of samples in water troublesome and time-consuming; with 0·1N-NH_3 as solvent and Britton and Robinson bu (pH 6 to 7) as diluent, soln. can be prepa quickly without detriment to stability or sub quent polarography. (*Cf.* Manoušek *et al., A Abstr.*, 1958, **5**, 2249.) P. BRYC

See also Abstracts—**1484**, Arsenic in orga matter. **1640**, Determination of piperazine. **16** Determination of beeswax in waxes. **1729**, De mination of cyanocobalamin. **1730**, Identificat of ascorbic acid. **1731**, Determination of to pherols.

Food

Foods and food additives, beverages, edible oils and fats, vitamins.

1716. **Technique for collection of food volatiles for gas-chromatographic analysis.** W. W. Nawar and I. S. Fagerson (Dept. of Food Technol., Univ. Mass., Amherst, U.S.A.). *Anal. Chem.*, 1960, (11), 1534–1535.—The technique described is continuous gas - liquid extraction ensuring high recovery of volatiles. The sweep gas, time of keeping and temperature depend on the sample. The technique has been used for the collection of volatiles from butter oil, maize oil, potato chips, cheese, shrimps and apple juice. C. B. Baines

1717. **Estimation of the available lysine in animal-protein foods.** K. J. Carpenter (Sch. of Chem., Univ. of Cambridge, England). *Biochem.* 1960, **77** (3), 604–610.—The reaction with 1-fluoro-2,4-dinitrobenzene for the determination of the free ε-amino-groups of lysine units in purified proteins is applied to animal-protein foods in an attempt to determine the extent of the nutritional damage that may occur through the formation of enzyme-resistant linkages with ε-amino-groups during processing. The dinitrophenyl proteins are hydrolysed with acid without separation from the digest, and the hydrolysates are extracted with ether. The measure of ε-dinitrophenyl-lysine in the aq. layer is based on the further decrease in colour in this layer after treatment with methyl chloroformate and re-extraction with ether.

J. N. Ashley

1718. **Gas chromatography in food analysis. I. Determination of the lower fatty acids in foods.** H. Diemair and E. Schams (Univ. Inst. f. Lebensmittelchemie, Frankfurt am Main, Germany). *Z. Lebensmitt Untersuch.*, 1960, **112** (6), 457–463.—The isolated acids can be satisfactorily separated in the Perkin - Elmer Fractometer (type 116) at 140° with a mixture of sebacic acid and sebacate as the stationary phase in a 2-metre column, and He (100 ml per min.) as carrier gas. A column of hydroxyethylene glycol is similarly used at 103° for the separation of the aromatic substances of the mes. A Hartmann and Braun electronic comparograph is used as detector. *Preparation of samples*: (*i*) *Milk*—Coagulate the sample (150 ml) with 5% H_2SO_4 and remove the fat and curd by centrifugation; make the aq. phase alkaline with $Ca(OH)_2$ soln., extract it with ethyl ether, and then evaporate nearly to dryness; acidify with HCl and extract the acids with ether; extract the acids into aq. Na_2CO_3 soln., evaporate the aq. phase to dryness, transfer the residue to a small tube, acidify, and extract the acids into ether (0·5 to 1 ml). (A similar alternative procedure involving a steam-distillation under reduced pressure is also described.) (*ii*) *Butter or cheese*—The sodium salts of the acids are obtained in aq. soln., free from fat, by a procedure analogous to that of the Röse - Gottlieb method; the subsequent treatment follows that described for milk. (*iii*) *Wine*—Extract the sample (100 ml) with ethyl ether - pentane (1 : 1) (3 × 100 ml); separate the acids from the neutral aromatic substances by shaking out with aq. 5% NaOH soln.; proceed with the aq. alkaline phase as described for milk. Dry the ether - pentane soln. as thoroughly as possible with anhyd. Na_2SO_4 and concentrate, if necessary, for the chromatographic examination. (*iv*) *Rye bread*—Extract the sample (10 g) with ethyl ether (100 ml); the dried ethereal soln. may be used for direct chromatographic examination or, if necessary, concentrated. Typical chromatograms are given for samples of milk, sherry and rye bread. P. S. Arup

1719. **Absorptiometric determination of fluoride in milk.** Report by Chemical and Metallurgical Services, U.K.A.E.A. (U.K.A.E.A., Risley, Warrington, Ches., England). U.K.A.E.A. Report PG 141(S), 1960. 8 pp.—The method involves curdling, evaporation and ashing of the milk, separation of F^- by steam-distillation, and absorptiometric determination of F^- by its bleaching effect on the aluminium complex of Solochrome Cyanine R (C.I. Mordant Blue 3) (**I**). *Procedure*—to 250 ml of sample warmed to 30° add cheese rennet (1 ml), stir until curdled and set aside for 30 min. Make alkaline to phenolphthalein with 4% NaOH soln. (< 5 ml), stir vigorously, set aside for 30 min. and decant the whey into a platinum dish and evaporate. Add the curd, evaporate to dryness and ash at 600°. Leach the ash with 10 ml of H_2O and transfer the contents of the dish to a 250-ml Claissen flask containing 2 g of Ag_2SO_4, rinsing the dish with 50 ml of H_2SO_4 (1 : 1). Steam-distil at 138° to 140° (liquid temperature), collecting 240 ml with the condenser dipping into 4% NaOH soln. (5 ml), and dilute to 250 ml. Adjust the pH of a 25-ml aliquot to 4·5 with HCl (1 : 10) or NaOH soln. (4%), add 2 ml of buffer (pH 4·5) (32 g of NH_4Cl, 220 ml of H_2O, 25 ml of glacial acetic acid and 7·5 ml of conc. aq. NH_3), check the pH, and add 15 ml of colour reagent. Dilute to 50 ml, heat in a water bath at 60° for 15 min., cool in cold water for 10 min. and set aside for 30 min. Measure the extinction at 535 mμ in a 1-cm cell against a reagent blank soln. distilled without the sample. Calibrate the spectrophotometer from 0 to 20 μg of F with standard NaF soln. *Colour reagent*—To 13·4 g of NH_4Cl in 450 ml of H_2O add 1 ml of glacial acetic acid, 20 ml of **I** (0·25% in 0·01M-HCl) and 10 ml of $Al_2(SO_4)_3.(NH_4)_2SO_4$ soln. (0·1867%), adjust the pH to 4·5 ± 0·02 with aq. NH_3 and dilute the soln. to 500 ml. Add 1 crystal of thymol, store for < 2 days in amber glass and filter before use. C. S. Howes

1720. **Determination of methoxychlor and/or metabolites in milk following topical application to dairy cows.** M. L. Cluett, W. K. Lowen, H. L. Pease and C. A. Woodhouse (Industrial & Biochemicals Dept., E.I. du Pont de Nemours & Co., Inc., Wilmington, Del.). *J. Agric. Food Chem.*, 1960, **8** (4), 277–281.—Methoxychlor was determined (*i*) by a method based on that of Fairing and Warrington (*Adv. in Chem. Ser.*, No. 1, 1950, 260), involving careful removal of fats, and reaction with H_2SO_4 to give a pink colour measured at 550 mμ, and (*ii*) by determining total organic Cl by solvent extraction of the milk, Pregl catalytic combustion of the extract residue, and spectrophotometric determination of the resulting Cl. Organic Cl-containing compounds other than methoxychlor were not detected.

M. D. Anderson

1721. **Rapid determination of mercury in apples by modified Schöniger combustion.** W. H. Gutenmann and D. J. Lisk (Pesticide Residue Lab., Dept. of Entomology, State Coll. of Agric., Cornell Univ., Ithaca, New York). *J. Agric. Food Chem.*, 1960, **8** (4), 306–308.—Loss of Hg by volatilisation during wet-ashing is avoided by using a modified Schöniger combustion. Apple tissue dried on Cellophane overnight *in vacuo* is burned in an

O-filled flask with a rubber-balloon attachment for pressure control. Combustion products are absorbed in 0·1N-HCl, and Hg is extracted by dithizone and determined spectrophotometrically. Recovery of 0·3 to 0·6 p.p.m. of Hg from apples averaged 83·6%. M. D. ANDERSON

1722. **Rapid method for determination of the husk content of barley and oats.** E. T. Whitmore (Plant Breeding Inst., Cambridge, England). *J. Inst. Brewing*, 1960, **66** (5), 407–408.—*Procedure*—The grain (20 g, of known moisture content) is mixed with 100 ml of H_2O containing 20 ml of NaClO soln. (10% of available Cl), NaOH (2·5 g) is added and the mixture is brought gently to the boil and boiled until the husk becomes detached and floats to the surface (approx. 3 min.). The husk is then decanted off, the kernels are thoroughly washed with H_2O, drained, spread on a filter-paper and dried in air overnight. They are then weighed, their moisture content is determined and the husk content obtained by difference. With barley, the results are lower than those by the method of Essery *et al.* (*Ibid.*, 1956, **62**, 150), but correlate well therewith. With oats, the results are higher than those by manual de-husking, and the correlation is not so good as with barley. It is claimed that the present procedure is more accurate than either of the older ones. R. E. E.

1723. **The E.B.C. scale of formazin haze units.** L. R. Bishop (The Brewery, Mortlake, London, S.W.14). *J. Inst. Brewing*, 1960, **66** (5), 388–389.—The Analysis Committee of the European Brewery Convention recommends the following procedure. Prepare a 1% aq. soln. of hydrazine sulphate, setting it aside for at least 4 hr. before use to ensure complete dissolution. Mix an aliquot of this soln. with an equal volume of 10% aq. hexamine soln. and set aside for 24 hr. to complete haze formation. This constitutes "formazin haze concentrate", and is stable for 3 months. Dilute the concentrate with water (1:10) to produce "100-unit E.B.C. haze dispersion", which is stable for 1 week. Suitable dilutions of this can be prepared with water (stable for 1 week) or beer (stable for 6 hr.). Such dilutions can be used for direct or instrumental visual haze comparisons, and for standardising various types of photo-electric instruments. The proportions of light scattered at different angles are similar for beer haze and for formazin haze (Thorne and Nannestad, *Ibid.*, 1959, **65**, 175). R. E. E.

1724. **Rapid method for iodine value and the iodimetric acid value.** W. Ruziczka. *Öst. ChemZtg*, 1960, **61** (10), 298–303 (in German).—The literature relating to the rapid iodine value method of Margoshes and co-workers (*Z. angew. Chem.*, 1924, **37**, 334), as applied to the analysis of unsaturated oils, resins, etc., is reviewed and the iodimetric method of determining acid value due to Kolthoff (*Chem. Weekbl.*, 1926, **23**, 260) is discussed. (37 references.) H. M.

1725. **Gas - liquid chromatography of fatty acid methyl esters: the "carbon number" as a parameter for comparison of columns.** F. P. Woodford and C. M. van Gent (Dept. Phys. Chem., Univ., Leyden, Netherlands). *J. Lipid Res.*, 1960, **1**, 188–190.—The retention times of at least 4 saturated straight-chain fatty acid esters are plotted on semi-logarithmic paper against chain length and a straight line joining the points is drawn. A value corresponding to the retention time of any other chromatographic peak can then be read from the graph, giving the chain length of the hypothetical saturated straight-chain ester which would be eluted at that point; that value is the "carbon number" of the ester on the particular liquid phase employed. Saturated esters have integral "carbon numbers" (*e.g.*, 15·0, 16·0 and 17·0); esters of branched-chain acids and unsaturated acids have not. The "carbon number" is characteristic of a particular ester on a particular liquid phase, and is sufficiently constant over small temp. ranges to allow comparison with other chromatograms, prepared with the same liquid phase in different laboratories. NUTR. ABSTR. REV.

1726. **Gas chromatography of unesterified fatty acids using polyester columns treated with phosphoric acid.** L. D. Metcalfe (Armour Industr. Chem. Co., Chicago, Ill., U.S.A.). *Nature*, 1960, **188**, 142–143.—A mixture (3 μl) of saturated and unsaturated fatty acids (C_{10} to C_{22}) can be separated effectively, in less than 1 hr., on a column (100 cm × 4 mm) of acid-washed Celite (60 to 80 mesh) coated with diethylene glycol adipate polyester (25%, w/w) containing 2% of H_3PO_4. The column temp. is 220° to 235°, the He pressure is 16 lb per sq. in., and a hot-wire detector is used. The peak areas are approx. proportional to the weight percentages of the free acids or their esters. The separation of stearic and oleic acids is incomplete. The methyl esters can be separated on the same column at 180°, only 1 μl of sample being required. W. J. BAKER

1727. **Esterification of fatty acids with diazomethane on a small scale.** H. Schlenk and J. L. Gellerman (Hormel Inst., Univ. of Minnesota, Austin, U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1412–1414.—A procedure is described for the preparation of small amounts (0·1 to 0·2 millimole) of diazomethane, which may be labelled with ^{14}C, from *N*-methyl-*N*-nitrosotoluene-*p*-sulphonamide. The diazomethane is carried by a stream of N into a soln. of the fatty acid in ether which contains 10% of methanol. Esterification is instantaneous and complete. G. BURGER

1728. **Paper chromatography of certain vitamins in phenol and butanol - propionic acid - water solvents.** E. L. Gadsden, C. H. Edwards and G. A. Edwards (Agric. and Tech. Coll. of N. Carolina, Greensboro, U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1415–1417.—The literature on chromatography of vitamins is reviewed. The migration of water-soluble and fat-soluble vitamins in two-dimensional chromatography on untreated paper has been studied with buffered water-saturated phenol as the first solvent and butanol - propionic acid - water as the second. The water-soluble vitamins were separated and located by detecting agents. Of the fat-soluble vitamins, only α-tocopherol was located. (42 references.) G. BURGER

1729. **Determination of vitamins and antibiotics by agar diffusion. I. Simplified computations for the plate method.** N. D. Ierusalimskiĭ, I. V. Konova and N. M. Neronova (Inst. Microbiol., Acad. Sci. USSR). *Mikrobiologiya*, 1959, **28** (3), 433–443.—The assay of vitamins and antibiotics by the plate method is discussed. It is found that if the recommended three replicate plates are used the mean error of the assay of cyanocobalamin is ± 7·3 ± 3·2% and that of the assay of streptomycin is ± 3·6 ± 2·1%. The use of four parallel plates has no appreciable influence on the accuracy of the

sults. A simplified method of computing the nount of the test substance is described, and bles are given that have been constructed for the mputation of vitamins and antibiotics.

II. Determinations of cyanocobalamin and its rivatives by paper chromatography. I. V. Konova, . M. Neronova, N. D. Ierusalimskiĭ and A. I. orisova. *Ibid.*, 1959, **28** (4), 490–494; *Ref. Zhur., him., Biol. Khim.*, 1960, (5), Abstr. No. 5783. — *rocedure*—Chromatograph the vitamin complex in ie test liquid, place the paper strips on agar eded with *Escherichia coli* 113–3, incubate and ien photograph the zones of growth on paper by ie contact method. Cut out the impressions of ie zones and weigh them. Determine the area by leans of the weight. [Knowing the weight of sq. cm of paper, calculate the mean diam. of the ne from the formula $D = 2\sqrt{S/\pi}$, where D is ie diam. and S is the area.] Prepare a corre- ponding nomogram beforehand. Calculate the nount of each component of the vitamin complex om the tables for the cup-plate method. A more eliable method is to add a pure preparation of a mponent to the soln. before chromatography. It shown that the cup-plate method and the iromatographic method are equally accurate.

K. R. Cook

1730. **Identification of ascorbic acid.** A. Gouds- aard (Rijks-Inst. Pharmacotherap. Onderz., eiden, Netherlands). *Pharm. Weekbl.*, 1960, **95** 20), 659–660.—The usual procedure for the identi- cation of ascorbic acid (**I**) with $FeSO_4$ is simplified. o a soln. of **I** (0·1 to 1%) are added $CaCO_3$ and $FeSO_4$. After shaking for 1 min. a violet colour is ormed. The sensitivity is 1 in 5×10^3 and the olour is fairly stable. Tartaric and oxalic acids nterfere and only a yellow colour is formed. urther, it was found that, in the iodoform reaction f Lieben, oxidised **I** is responsible for the pink olour which occurs after addition of iodine and aOH. The colour intensity reaches its maximum fter 30 min., and the colour is then very stable. Iaximum absorption is at 510 mμ. The reaction an be used for the identification of **I** in multi- itamin preparations and in the presence of salicylic cid derivatives. M. J. Maurice

1731. **Studies on vitamin E. IV. Simultaneous eterminations of tocopherols, ubiquinones and ibichromenols (substance SC) in animal tissues: econsideration of the Keilin - Hartree heart prepara- ion.** A. T. Diplock, J. Green, E. E. Edwin and . Bunyan (Walton Oaks Exp. Sta., Vitamins Ltd., adworth, Surrey, England). *Biochem. J.*, 1960, **6** (3), 563–571.—The method of analysis is based n that of Edwin *et al.* (*Anal. Abstr.*, 1960, **7**, 4506) or tocopherols with several modifications. The issue (20 g) is homogenised with Na_2SO_4 - acetone t − 70° and extracted for 3 hr. in the dark with cetone. The lipid extract is saponified with aturated aq. KOH - ethanol containing pyrogallol nd the hydrolysate is extracted with ethyl ether. An aliquot (10%) of the non-saponifiable extract nay be removed at this stage, if required, for the letermination of vitamin A by the $SbCl_3$ method. The remainder of the extract is dissolved in nethanol and sterols are removed at − 15°. The esidue, after evaporation of the methanol, is issolved in light petroleum (b.p. 40° to 60°), and the soln. is passed through a column of Decalso F floridin earth is unsuitable). The column is eluted with benzene, and two-dimensional paper chroma- tography is carried out with 25 or 50% of the eluate as described by Edwin *et al.*, except that 95% (v/v) ethanol is used as the mobile phase for the second dimension. The papers are dried in air, and the spots are observed under u.v. light. Another chromatogram is sprayed heavily with a fresh $FeCl_3$ - 2,2′-dipyridyl reagent to indicate the positions of tocopherol and ubichromenol. The soln. and papers should be shielded as far as possible from daylight. Each clearly separated spot is cut out and eluted. Tocopherols are eluted with 0·07% (w/v) 2,2′-dipyridyl in ethanol, and are determined by the usual method. Ubichromenols, if distinct, are eluted and determined with $FeCl_3$. Combined ubiquinone - ubichromenol spots are eluted with ethanol; one-half of the eluate is used for the determination of ubichromenol, and the other half for the determination of ubiquinone by the borohydride method of Crane *et al.* (*Biochem. Biophys. Acta*, 1959, **32**, 73). Tocopherylquinone and vitamin K_1 are only rarely present, and can be determined by their u.v. spectra. J. N. Ashley

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See also Abstracts—**1482**, Polyphosphates in meat products. **1484**, Arsenic in org. matter. **1577**, Nitrogen in org. materials. **1659**, Destruction of org. matter. **1693**, Cystine in foods. **1756**, Dieldrin in animal fats. **1766**, Hydrometers for milk.

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

1732. **Sensitive device for indicating contamination of the air.** B. Kamieński (Inst. of Phys. Chem., Acad. of Sci., Kracow, Poland). *Electrochim. Acta*, 1959, **1**, 278.—The apparatus consists of a very thin (of the order of the mean free path of gas molecules) hydrophilic film containing a colloidal electrolyte, covering the cross-section of a platinum wire (0·01 to 0·9 mm in diam.) melted into a glass rod and joined electrically to a reference electrode. The potential arising from such a device is measured with a valve voltmeter or (better) with a quadrant electrometer, since the internal resistance is of the order of 10^9 to 10^{12} ohms. Very low concn. of a pollutant affect the potential considerably; *e.g.*, a concn. of 0·000007% of Cl in the air causes a shift of 40 mV; the changes are reversible only over a limited concn. range of the contaminant. Organic colloidal electrolytes give films of longer mean life; greater sensitivity is attained with inorganic ones. From a practical standpoint, soap films give good results.

J. Electroanal. Chem. Abstr.

1733. **Spectrographic determination of beryllium in the atmosphere.** M. S. W. Webb (A.E.R.E., Woolwich Outstation, London, England). *Atom*, 1960, (45), 20–31.—Methods for the spectrographic determination of Be in air are reviewed. The usual method is to filter the air and analyse the filter-paper. The effects of the sampling rate, filter medium, and various methods of analysis are discussed. The sampling rate should not be less than the normal breathing rate of man, and the filter medium should capture the clinically important particles of radius 0·4 to 0·8 μ. The effects of filtration velocity on percentage penetration of filter-papers are studied. Glass-fibre papers would seem to be good for this purpose, but unfortunately they yield a beryllium blank, and this must be eliminated for the determination of small amounts of Be. In the analysis of the filter-paper, the best

method is to impregnate it with ferric nitrate soln. and ash it at low temp.; the resulting ferric oxide serves both as a carrier for Be and as a spectrographic buffer. The method is further improved by using photo-electric recording in place of the lengthy photographic process and using a triggered a.c. arc for better Be excitation. Solution methods of analysis of filter-papers are briefly discussed. The disadvantage of these methods is that they require time, and the Churchill - Gillieson monitor, briefly described, was designed for immediate Be monitoring. However, it also has disadvantages: its response varies with particle size, and it has no provision for automatic calibration. A new monitor was therefore designed (called the Woolwich monitor) for analysis within 1 min. after sampling, and it is described in detail. The inefficiency of excitation of condensed sparks was overcome by a modified triggered a.c. arc, the behaviour of which is similar to that of a d.c. plasma arc; its circuit is given together with that for the recording system. Calibration is provided for by means of a spark discharge between a copper and a copper - beryllium electrode, evolving Be at a constant rate. The performance of the monitor was evaluated for beryllium aerosols of narrow particle-size ranges; high concn. of Be, of too short a duration to be detected by filtration techniques, were detected by this monitor. NUCL. SCI. ABSTR.

1734. **Morin method for the detection of beryllium in air samples.** J. Walkley (Mass. Inst. of Technol., Cambridge, U.S.A.). *J. Amer. Ind. Hyg. Ass.*, 1959, (20), 241–245.—The fluorimetric determination of Be in air with morin, sensitive to 0·004 μg of Be or 0·02 μg per sample, is described. One ml of 0·05M-$Al(NO_3)_3$ and 1 ml of 25% NH_4Cl soln. are added to a 10-ml aliquot of a prepared sample. A drop of phenol red is added, and the soln. is made alkaline with 3N-aq. NH_3 and centrifuged for 10 min. The resulting ppt. is dissolved with 1 ml of 4N-NaOH; 2 drops of 10% KCN soln. are added, and the soln. is diluted to 10 ml. After centrifuging, 1 ml of dil. morin soln. is added, and the fluorescence is measured immediately against a quinine sulphate standard. An aliquot containing 0·01 μg of Be shows no interference at a 100-μg level of Ca, Cu, F, Fe, Mg, PO_4^{3-}, Ti, U or Zr; Zn does not interfere at a level of 75 μg.
CHEM. ABSTR.

1735. **Method for identifying particulate fluoride compounds.** B. J. Tufts (Dept. of Meteorology, Univ. Chicago, U.S.A.). *Anal. Chim. Acta*, 1960, **23** (3), 209–214 (in English).—The method is intended for the identification of air-borne dust particles collected on a membrane filter. The filter is floated on the reagent soln. [0·057M in Pb, made by mixing 1 part of 10% aq. NaCl soln. with 3·2 parts of 10% aq. $Pb(NO_3)_2$ soln., centrifuging and using the supernatant liquid] until wetted thoroughly. It is then transferred to the surface of water for 2 min., then dried and mounted. With fluorosilicates the pH of the reagent is reduced to 2·5 with acetic acid, and the reagent is soaked on to blotting paper. This gives more compact ppt. In each case the specimen is examined microscopically and the diameters of the crystalline aggregates are measured. The diameter of the ppt. is a measure of the size of the particle which formed it, and calibration graphs are presented for NaF and K_2SiF_6; SO_4^{2-} and PO_4^{3-} give ppt., but these may be recognised by the use of polarised light. Ppt. of F^- and PO_4^{3-} show polarisation colours, whereas those of SO_4^{2-} do not. The PO_4 colours are less intense, and can be distinguishe with practice. H. M.

1736. **Determination of micro amounts of chlorin in air.** E. P. Bŷkov, A. K. Pankova, A. I Khlŷstov and M. N. Stepochkina. USSR Pat. N 127,070 (10.3.1960).—The air is drawn at a rate ≃ 1·5 litres per min. per sq. cm through a pape strip that has been impregnated with a 20% sol of choline iodide in 90% methanol and dried; is quant. absorbed, forming a yellow spot, th colour of which is compared with standards. Th spot may be cut out and treated with 1 ml water, and the colour of the soln. compared in microcolorimeter with standard soln. of iodine i 1% choline iodide, containing 0·01 to 1·0 mg iodine per ml. With low Cl contents, add to th soln. a 0·1% methanolic soln. of *p*-amino-*NN* dimethylaniline (2 drops per ml of soln.), an compare the resulting red colour with that of sol containing 0·0005, 0·001, 0·005 or 0·01 mg iodine per ml, or with permanent standards pre pared from soln. of fuchsine in 0·005N-H_2SO_4. Th standard soln. of iodine in 1% choline iodide ar stable for a month. C. D. KOPKIN

1737. **Method for the determination of tar hydrocarbons in air.** E. Malý (Inst. Hyg. and Ind Diseases, Bratislava, Czechoslovakia). *Pracovn Lékařství*, 1960, **12** (7), 347–354.—Liquid paraffi on Whatman No. 4 paper was used as stationar phase and methanol satd. with liquid paraffin a the mobile phase for the ascending chromatographi separation of the 3,4-benzopyrene fraction fro other fractions. The semi-quant. determinatio of 3,4-benzopyrene, separated from pyrene an 1,2-benzopyrene, has been carried out by th planimetric method. The sample of air is draw through a tube containing three columns of cotto wool moistened with liquid paraffin; the tube i washed out with $CHCl_3$, the $CHCl_3$ is then evaporate to 0·05 to 0·2 ml and placed on the chromatographi paper by means of a Linderström - Lang pipette.
J. ZÝKA

1738. **A new method for the continuous analysi of dissolved oxygen in water.** J. M. Wright an W. T. Lindsay, jun. (Westinghouse Electric Corp. Pittsburgh). *Proc. Amer. Power Conf.*, 1959, **21** 706–721.—In the apparatus described, a packe bed of thallium metal is used for the continuou determination of dissolved oxygen in water Thallium is not attacked by water or dissolve gases other than oxygen. With oxygen it form thallous oxide which is highly soluble in water thus increasing the specific conductance of th water. The performance of test and prototype units is described and the results are compare with those of Winkler analyses. More precis measurements are required to confirm the accuracy of the device at concentrations below 0·05 p.p.m The method appears promising for measurement i the parts per 10^9 range and, perhaps, even lowe with suitable modifications. NUCL. SCI. ABSTR.

1739. **Methods for separation and determination of beryllium in sediments and natural waters.** J. R Merrill, M. Honda and J. R. Arnold (Dept. Chem. Univ., Princeton, N.J., U.S.A.). *Anal. Chem.* 1960, **32** (11), 1420–1426.—The Be in sea water (5×10^{-13} g per ml) was concentrated by passage through Dowex 50-X8 resin (200 to 400 mesh) the Fe^{3+} or Mn^{3+} form of which had been treated with excess of aq. NH_3, and by elution of Fe with

M-oxalic acid, followed by elution with 6M-. (to remove the Be). The Be was then determined spectrophotometrically as the acetylacetone plex (absorption max. at 295 mμ): 0·2 μg of can thus be reliably determined. The method also applicable to ocean-bed and other sedintary samples, and to the isolation of Be for measurement of ^{10}Be. J. P. STERN

1740. **Determination of radioactive strontium in er samples.** V. P. Shvedov, T. P. Makarova, M. Ivanova and N. A. Pavlova. *Radiokhimiya*, 9, **1** (5), 616–618; *Ref. Zhur., Khim.*, 1960, (12), str. No. 45,983.—Treat the dry residue (≃ g) from evaporation of the sample (50 to 100 es), together with carriers, with a mixture of ml of HCl (sp. gr. 1·19), 50 ml of HNO_3 (sp. gr. 8) and 125 ml of water, with warming. Evapoe the soln. to dryness, dry the residue for 1 hr. 105° to 110°, dissolve it in 250 ml of 6N-HCl, l evaporate the soln. to dryness; dry the residue before, and dissolve it by heating for 1 hr. h 200 ml of 0·5N-HCl. Centrifuge off the oluble residue (containing Si and part of the and Ba) from the soln., which contains the in bulk of Sr, Ba, Ca and other elements, treat with H_2SO_4 - HF to remove SiO_2, evaporate the d soln. to dryness, fuse the dry residue with $_2CO_3$, extract the melt with water, and dissolve $BaCO_3$ and $SrCO_3$ in HCl. Add the soln. to a ilar soln. obtained by pptg. the main bulk of Sr, Ba and Ca as sulphates in the presence of anol, converting into carbonates and dissolving HCl. Precipitate Ba from the combined soln. $BaCrO_4$, and separate the Sr from Ca by salting t as $Sr(NO_3)_2$ five times with HNO_3 (sp. gr. 1·45 1·46). Further purify the Sr by adding Y as rrier, pptg. as oxalate, and igniting to SrO. e activity of the ^{90}Sr is determined by measuring e activity of the daughter ^{90}Y. The hydroxide Y is purified from Sr by pptg. the Sr as the rate, then pptg. Y as the oxalate and igniting Y_2O_3. The activity of ^{89}Sr and ^{90}Sr is determned by recording the decay curve and analysing by the rectification method [*Ref. Zhur., Khim.*, 56, (15), Abstr. No. 45,931]. Methods for the nultaneous determination of the fission products , Sb, etc., present in the samples are briefly scribed. C. D. KOPKIN

1741. **Spectrophotometric determination of ioride in water.** F. I. Brownley, jun., and C. W. owle, jun. (Clemson College, Clemson, S.C., S.A.). *Anal. Chem.*, 1960, **32** (10), 1330–1332.—he method proposed is based on the bleaching fect of F^- on the Th^{IV} - phenylfluorone complex pH 3·6. Extinctions are measured at 530 mμ. otential cationic interferences are removed by assage through a column of Amberlite IR-120 H^+ form). Chloride does not interfere up to 00 p.p.m., but PO_4^{3-} interfere seriously, and if resent in concn. > 1 p.p.m. must be separated distillation. The method is applicable to F^- ncn. of 1 to 5 p.p.m. T. R ANDREW

1742. **Determination of iodine in mineral waters.** V. Rastegina. Publ.: "Thyroid Disease", Kiev, osmedizdat, Ukr.SSR, 1959, **2**, 275–278; *Ref. hur., Khim.*, 1960, (18), Abstr. No. 73,138.—The ethod is based on extraction of the iodine by recipitation chromatography, its desorption as $_2HgI_4$, and colorimetry of the complex HgI_2.CuI. *rocedure*—Make the sample alkaline to phenolthalein with K_2CO_3, evaporate on a sand bath to 50 ml, filter, wash the residue several times with water, and make the filtrate and washings up to 100 ml. Pass 1 ml of the resulting soln. through a column (8 cm × 0·6 cm) filled with Al_2O_3 (Brockmann) impregnated with $Hg(NO_3)_2$. The resulting mercury halide zones lie in the order HgI_2, $HgBr_2$ and $HgCl_2$. Wash the column with water until the wash-liquor is free from Cl^-; the $HgCl_2$ and $HgBr_2$ are desorbed, while the HgI_2 zone is displaced slightly downwards. Dissolve the ppt. of HgI_2 in anhyd. ether, evaporate the solvent, and dissolve the residue in excess of KI soln. Place drops of the resulting soln. on filter-paper impregnated with a suspension of CuI, and determine the iodine content by comparing the intensity of the orange - red HgI_2.CuI complex with standards. C. D. KOPKIN

1743. [**Sludge-digestion**] **gas analysis by chromatography.** W. L. Henderson. *Wat. & Sewage Wks*, 1960, **107** (8), 312–313.—Digester gas is chromatographed on a silica-gel column with He as carrier gas; O and N are not separated, but this is usually immaterial for this work. A thermal-conductivity detector is employed. The use of the method to follow CO_2 concn. is described. S.C.I. ABSTR.

1744. **Colorimetric determination of phenolic materials in refinery waste waters. Removal of sulphides by silver nitrate.** G. E. Gordon (Manufacturing Dept., Standard Oil Co. of California, Richmond, U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1325–1326.—The 4-aminophenazone method of the American Petroleum Institute has been modified to eliminate the phenol distillation stage. Sulphides are removed by pptn. with $AgNO_3$ and the excess of Ag is pptd. with NaCl. The results show good agreement with those obtained by the distillation method, are obtained in half the time and are thought to be more accurate. A. R. ROGERS

1745. **Determination of β-activity of liquid wastes.** F. Běhounek and V. Zelenková (Fac. Tech. and Nuclear Physics, Prague). *Jaderná Energie*, 1960, **6** (9), 299–302.—The accuracy of the conventional method for the determination of the β-activity of waste waters with an end-window Geiger - Müller tube was increased by the use of double filtration. The determination of the activity of mixtures of radio-isotopes with very different β spectra is thus possible as well as the detection of ^{90}Sr and ^{90}Y. J. ZÝKA

See also Abstracts—**1425**, Decaborane in air. **1495**, Hydrogen sulphide in air. **1504**, Control of chromate in waste liquors. **1625**, Aliphatic aldehydes in air. **1639**, Pyrene deriv. in air.

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

1746. **X-ray fluorescence determination of zinc in plant tissue.** L. D. Whittig, J. R. Buchanan and A. L. Brown (Univ. of California, Davis, U.S.A.). *J. Agric. Food Chem.*, 1960, **8** (5), 419–421.—It has been shown that inconsistent results for Zn are obtained when the intensity of the Zn emission maxima is taken as a function of the Zn concn. in plant tissue of complex and variable composition. For results of high accuracy, known amounts of Zn are added to the dried plant sample, and the amount of Zn in the sample is calculated

on the basis of the increase in emission-to-scatter ratio, or on the increase of actual emission intensity per unit of Zn added. R. A. HENDEY

1747. **Rapid method for determining adsorbed sodium and potassium in carbonate soils.** K. Sh. Shatemirov. *Pochvovedenie*, 1960, (2), 107–109; *Ref. Zhur., Khim.*, 1960, (18), Abstr. No. 73,036.—Treat the sample of soil (5 g) with 0·2N-oxalic acid (100 ml × 5), decant the soln., filter and evaporate. Moisten the dry residue in a platinum or nickel dish, evaporate to dryness, heat on a sand bath (to remove excess of oxalic acid) and ignite at 450° to 500° for 2 to 2·5 hr. to convert alkali-metal and alkaline-earth oxalates into carbonates, and iron and aluminium oxalates into oxides. To avoid dissolution of Mg, the alkali-metal carbonates are extracted with satd. aq. MgO soln. (**I**). Filter off the residue, wash it with **I** and dilute the filtrate with **I** to 200 or 500 ml. Titrate an aliquot of the soln. with 0·02N-H_2SO_4 to methyl orange, titrate **I** similarly, and obtain the content of Na and K by difference. The relative error is ± 9% as compared with the gravimetric determination with zinc uranyl acetate and ± 10% as compared with the Gedroits method. C. D. KOPKIN

1748. **Simultaneous determination of phosphate and sulphate ions in the presence of metal contaminations [in fertilisers].** L. Szekeres and E. Bakács-Polgár (Inst. of Chem., Univ. of Agric. Sci., Budapest, Hungary). *Magyar Kém. Lapja*, 1960, **15** (10), 460–462; *J. Agric. Food Chem.*, 1960, **8** (5), 417–419.—A volumetric method is described. The sample is dissolved in boiling HCl, and Fe^{3+} and Al^{3+} are masked with EDTA (disodium salt) and Cu^{2+} and Mg^{2+} with 1,2-diaminocyclohexane-tetra-acetic acid, both in alkaline soln. The excess of complexan is then destroyed by the addition of $MgCl_2$ soln., with Eriochrome black T as indicator. Ethanol is then added to give a 10% soln. and the phosphate is determined by titration with 0·1M-$MgCl_2$. The ethanol content is then raised to 40% and the sulphate is determined by titration with 0·1M-$BaCl_2$. The results obtained are compared with those given by gravimetric methods. The effects of Ca^{2+}, Mg^{2+}, Al^{3+}, Fe^{3+} and F^- are investigated. B. VASZKO

1749. **Analysis of MCPA/TBA herbicide formulations. I. A liquid - liquid chromatographic method for determination of 2,3,6-trichlorobenzoic acid.** K. Gardner and K. C. Overton (Fisons Pest Control Ltd., Cambridge, England). *Anal. Chim. Acta*, 1960, **23** (3), 271–281 (in English).—With this method, 2,3,6-trichlorobenzoic acid (TBA) can be determined in the presence of all other likely chlorobenzoic acids and chloromethylphenoxyacetic acids. The mobile phase is isopropyl ether, the support is Hyflo Super-Cel, and the stationary phase is a buffer made from 1 vol. of 2M-aq. K_2HPO_4 and 1·5 vol. of 2M-NaH_2PO_4. The sample, containing 2 g of acids, is dissolved in water, acidified with HCl, and extracted several times with ethyl ether. The ether extracts are combined and made up to 200 ml; 1 ml is transferred to the column and washed just into the column with the mobile phase. The mobile phase is then passed at the rate of 2 ml per min. (using pressure of N) and 60 × 5-ml fractions are collected, followed by 2 × 100-ml fractions. Each fraction is titrated with 0·01N-Na methoxide in isopropyl ether, with a specially prepared indicator based on bromothymol blue. The required fractions are Nos. 28 to 60, which will contain one small and one large compone The titre of the latter is a measure of the T content. The acids contained in the other fracti are listed.

II. A gas - liquid chromatographic method the determination of 4-chloro-2-methylphenox acetic acid. K. Gardner and K. C. Overton. *Ib* 1960, **23** (4), 337–345 (in English).—After esterifi tion under conditions that prevent esterification the halogen-substituted benzoic acid impurities, t sample is extracted with dichloromethane, a kno quantity of ethyl benzoate is added as an inter standard and the mixture is evaporated in a wa bath at 80°. A small aliquot (3 μl) is transfer to a Perkin-Elmer Fractometer having a colu of Celite 545 and Apiezon M, and the chroma gram is prepared at a column temp. of 190° wi H as carrier gas (50 ml per min.). A chromatogra is reproduced that illustrates the separation of eth benzoate, 2-methylphenoxyacetic acid, dichlo benzoic acids, 6-chloro-2-methylphenoxyace acid, 4-chloro-2-methylphenoxyacetic acid, 2,3, and 2,4,5-trichlorobenzoic acids (one peak), 2,3, trichlorobenzoic acid and 4,6-dichloro-2-meth phenoxyacetic acid. H. M. T. R. ANDREW

1750. **Determination of α-(4-chloro-2-meth phenoxy)propionic acid in chloromethylphenox propionic acids by gas - liquid chromatography wi an internal standard.** H. G. Higson and D. But (Lankro Chemicals Ltd., Salters Lane, Eccl Manchester, England). *Analyst*, 1960, **85**, 65 663.—A selective herbicide formulation is bas on CMPP, which is a mixture of chlorometh phenoxypropionic acids containing α-(4-chloro-methylphenoxy)propionic acid (**I**) as the acti ingredient with various proportions of 6-chloro-methyl-, 4,6-dichloro-2-methyl- and 2-meth phenoxypropionic acids. To convert the acids in a form suitable for gas - liquid chromatograph they are extracted from acid aq. soln. with CH and converted into their butyl esters with butanol and dry HCl by a specified procedur The gas - liquid chromatographic apparatus described. Dimethyl phthalate is used as a internal standard. The method of calculating t content of **I** from the areas under the sample pea and standard peak is described. A modified pr cedure is given in which an internal standard not required. A. O. JONES

1751. **Micro-coulometric gas chromatography pesticides.** D. M. Coulson, L. A. Cavanagh, J. de Vries and B. Walther (Dept. of Chem., Stanfo Res. Inst., Menlo Park, Calif., U.S.A.). *J. Agri Food Chem.*, 1960, **8** (5), 399–402.—A new rap screening method is described. The apparat comprises a chromatography column, a micr combustion furnace, a power supply for the chrom tograph and a coulometer. The chromatograph column used was an aluminium tube (0·25 in. b 6 ft.) packed with 30 to 60-mesh Chromoso (washed with 6N-HCl, and then H_2O) coated wit 15 to 30% (w/w) of Dow - Corning high-vacuu silicone grease (extracted first with ethyl acetate The coulometer system is essentially that Coulson and Cavanagh (*Anal. Chem.*, 1960, **32**, 124 with a more sensitive detector. The chromato graph operates in the conventional manner an effluent gases are mixed with O, burnt in th furnace, and bubbled through the soln. in th titration cell. The resulting Cl or S is coul metrically titrated with an automatic titrato

mixed pesticides the time required for each pound to pass through the column must be viously determined, in order to identify the ponents. The method is rapid (≃ 1 hr.) and be used to determine many pesticides (*e.g.*, achlorocyclohexane, aldrin, dieldrin, DDT, rdane, endrin, toxaphene and other chlorinated anic pesticides) in a single operation. Phosphoro-ates (*e.g.*, malathion, parathion and Systox) also be determined. R. A. Hendey

1752. **Separation and estimation of the four ecticidal constituents of pyrethrum, *Chrysanthemum cinerariaefolium*, by elution chromatography on a column of absorptive charcoal.** H. henson (Pyrethrum Board of Kenya, Nakuru, nya). *Pyrethrum Post*, 1960, **5** (4), 22–30.—e four biologically active ingredients of pyre-um are separated by elution from a charcoal umn with light petroleum - ethyl ether (2:3) as vent. The spectrophotometric evaluation of the ctions is discussed and the following constants reported—$\lambda_{max.}$ (in light petroleum and in anol), cinerin I, 200 mμ and 225 mμ; pyrethrin 221 mμ and 224 mμ; cinerin II, 229 mμ and mμ; and pyrethrin II, 227 mμ and 228 mμ; culated values for ϵ_{227} (in ethanol) were 19,300, 400, 25,800 and 46,600, respectively. The thod is tedious and not suitable for routine plication and is affected by the presence of ergists, surface-active agents and other addi-es. A sample of commercial pyrethrum extract taining 25% of total "pyrethrins" ("Methods Analysis," A.O.A.C., 8th Ed., 1955) gave a mean 23·15% of total "pyrethrins", with a coeff. of riation of 1·81% (13 determinations). The sum cinerin I and pyrethrin I is close to that obtained the A.O.A.C. method for "pyrethrins I", but the m of cinerin II and pyrethrin II is ≃ 85% of e A.O.A.C. "pyrethrins II" result.
H. B. Heath

1753. **Analysis of crop extracts for traces of orinated pesticides by gas - liquid partition romatography.** E. S. Goodwin, R. Goulden, A. hardson and J. G. Reynolds (Woodstock Agric. s. Centre, "Shell" Research Ltd., Sittingbourne, nt, England). *Chem. & Ind.*, 1960, (39), 1220–21.—Aldrin and dieldrin in millimicrogram ounts have been detected without prior "clean-" of the crop or tissue extract. An argon detector used with a low applied potential, and negative aks, characteristic of electron capture, are shown these chlorinated compounds. With a 6-ft. umn of 10% (w/w) of E301 silicone on kieselguhr 230°, and a flow of Ar of 6 litres per hr., the tection limits are 0·05 p.p.m. of aldrin and p.p.m. of dieldrin. *pp'*-DDT and γ-BHC show similar order of detectability, but tend to be graded on the column, as does endrin, which ves multi-peak chromatograms. R. M. S. Hall

1754. **Cryoscopic determination of the purity of dane.** R. Handley (Nat. Chem. Lab., Tedding-n, Middx., England). *J. Appl. Chem.*, 1960, **10**, 353–357.—A simple freezing-point apparatus described. *Procedure*—Powdered lindane (25 g) melted in the apparatus and dried by the passage a stream of dry N for 1 hr. During cooling, the mp. is read at 1-min. intervals and the freezing-int is determined. The purity of the sample, in ms of its γ-BHC (**I**) content, is calculated from e observed freezing-point and the freezing-point d cryoscopic depression of pure **I**. Collaborative sults from four laboratories show good agreement. The accuracy is ± 0·1% and results agree with those obtained by the differential refractometric method. A. C. R. Hartley

1755. **Residue analysis of a chlorinated insecticide (Thiodan) by combination of gas chromatography and infra-red spectrophotometry.** G. Zweig, T. E. Archer and D. Rubenstein (Pesticide Residue Res., Univ. of California, Davis, U.S.A.). *J. Agric. Food Chem.*, 1960, **8** (5), 403–405.—Thiodan (**I**) was used to develop a general method of trace analysis for chlorinated pesticides. The chromatographic column used was a stainless-steel column (0·25 in. by 6 ft.) packed with Dow - Corning No. 11 high-vacuum grease (30% w/w) on 35 to 80-mesh Chromosorb. The column temp. was 225° and the carrier gas was He at a rate of 167 ml per min. Under these conditions the effluent time for **I** was 4 to 7·2 min. The effluent stream was collected in a tapered glass tube containing a pad of glass wool soaked in CS_2, then the tube was washed well with CS_2 (25 ml), and the CS_2 evaporated. The residue (containing 4·8 to 120 μg of **I**) was re-dissolved in CS_2 (0·2 to 5 ml), transferred to a micro-cell (capacity 0·2 ml) for i.r. spectrophotometry, and scanned between 8 and 8·5 μ, with pure CS_2 for the reference beam. The absolute extinction at 8·37 μ was measured and calculated by using the base-line method. Crop material (lucerne, peaches and pears) was extracted (with the roller-stripping technique) and then subjected to the procedure described above. Recoveries of added **I** were 86·4 to 115%, and the absolute sensitivity for **I** was 4 μg per ml. The advantages of the method are simplicity and the simultaneous qual. and quant. analysis of the compounds under investigation. R. A. Hendey

1756. **Colorimetric determination of dieldrin and its application to animal fat.** C. Cueto, jun. (Communicable Disease Center, Bureau of State Services, Public Health Service, U.S. Dept. of Health, Education and Welfare, Phoenix, Ariz.). *J. Agric. Food Chem.*, 1960, **8** (4), 273–276.—Dieldrin is determined in μg amounts by treatment with diphenylamine in the presence of $ZnCl_2$ to yield a purple colour, which is measured in acetic acid soln. at 650 mμ. The method is applied to dieldrin in animal fat by alkaline hydrolysis of the fat, extraction with hexane, chromatography of the extract on alumina columns to remove impurities, evaporation of the eluate to dryness, and addition of the diphenylamine and $ZnCl_2$ reagents. The alkaline hydrolysis destroys other chlorinated insecticides that might interfere.
M. D. Anderson

1757. **Determination of toxaphene by a spectrophotometric diphenylamine procedure.** A. J. Graupner and C. L. Dunn (Res. Center, Hercules Powder Co., Wilmington, Del., U.S.A.). *J. Agric. Food Chem.*, 1960, **8** (4), 286–289.—Toxaphene is fused with diphenylamine in the presence of $ZnCl_2$ to give a greenish-blue product which is measured in acetone or acetic acid soln. at 640 mμ. Clean-up procedures for hexane extracts include (*i*) chromatography on a Florisil column, for extracts containing little fat, (*ii*) partitioning between hexane and H_2SO_4 - fuming H_2SO_4, for extracts containing acid-sensitive material, and (*iii*) extraction with dichloromethane - H_2SO_4, for extracts containing much fat, followed by transfer of the toxaphene to hexane, and application of process (*i*). Toxaphene is extracted from formulations with boiling

methanol. Some other chlorinated insecticides may interfere. M. D. Anderson

1758. **Colorimetric determination of Guthion residues in crops.** W. R. Meagher, J. M. Adams, C. A. Anderson and D. MacDougall (Chemagro Corp., Kansas City, Mo., U.S.A.). *J. Agric. Food Chem.*, 1960, **8** (4), 282–286.—Guthion is separated (*a*) from cotton-seed oil by dissolving the sample in light petroleum, extracting with methyl cyanide and chromatographing on alumina; (*b*) from fruit by macerating the sample with acetone, filtering, adding HCl, and extracting with $CHCl_3$; (*c*) from cole crops by extracting the sample with isopropyl alcohol, and chromatographing on a column of mixed adsorbents; and (*d*) from milk by blending the sample with acetone, filtering, re-extracting the acetone-insol. material with benzene, combining the acetone and benzene extracts, evaporating the solvents, and partitioning the residue between hexane and methyl cyanide; the pesticide passes into the methyl cyanide. Guthion is converted by alkaline hydrolysis into anthranilic acid, which is diazotised, coupled with *N*-1-naphthylethylenediamine dihydrochloride, and measured at 555 mμ. The oxygen analogue of Guthion produces 50 to 70% of the expected colour. The method can also be used for the ethyl homologue of Guthion.
M. D. Anderson

1759. **Triphenyltin compounds in crop protection and the determination of residues.** E. Kröller (Bundesgesundheitsamt, Max von Pettenkofer-Inst., Berlin-Dahlem). *Dtsch. LebensmittRdsch.*, 1960, **56** (7), 190–193.—The method involves extraction with dichloromethane, phasic separation of triphenyltin compounds and their water-soluble decomposition products, and determination of Sn after destruction of organic matter and distillation of the Sn as tetrabromide. *Phasic separation*—Cautiously shake an aliquot of the filtered dichloromethane extract (50 ml) with 5% EDTA (disodium salt) soln. (10 ml) and commercial Zephirol soln. (2 ml) and allow to separate. Wash the aq. phase with dichloromethane (10 ml) and shake the combined dichloromethane extracts with 0·5% EDTA (disodium salt) soln. (10 ml). *Distillation of Sn*—To the soln. remaining from the destruction of organic matter with H_2SO_4, HNO_3 and satd. $KMnO_4$ soln. add 50% H_2SO_4 (10 ml) and hydrazine sulphate (1 to 2 g) and distil at 145° to 160° in a current of N by slow addition of a mixture of 6N-HCl (15 ml) and 40% HBr (8 ml) and collect the distillate under water. After removal of Br from the distillate, determine Sn by conventional procedures, *e.g.*, either polarographically or colorimetrically with dithiol. E. C. Apling

1760. **Modifications to a colorimetric method for determining dichlorophen [on textiles].** F. C. Ashton (Min. of Aviation Chem. Inspectorate, H.Q. Building, Royal Arsenal, Woolwich, London). *Analyst*, 1960, **85**, 685–686.—Application of the test described by Gottlieb and Marsh (*Brit. Abstr. C*, 1946, 118), which involves the use of 4-aminophenazone with $K_3Fe(CN)_6$ as oxidising agent, to the determination of dichlorophen (5,5′-dichloro-2,2′-dihydroxydiphenylmethane) (**I**) on textiles showed that $K_3Fe(CN)_6$ is an unsatisfactory oxidising agent for this purpose, but that KIO_4 is satisfactory. Small pieces of the textile sample (1 or 2 g) are extracted with boiling $Na_2B_4O_7$–NaOH buffer soln. (pH 9·7) (4 × 25 ml). The combined extracts are adjusted to 200 ml with the buffer soln. A 2-ml aliquot of the filtered liquid is diluted to $\simeq$ 30 ml with the buffer soln., a 1·5 ml of 1% aq. 4-aminophenazone soln. is adde followed by 10 ml of 0·25% aq. KIO_4 soln. T mixture is diluted to 50 ml with the buffer sol and the extinction is measured at 510 mμ again a reagent blank and referred to a calibration grap prepared with a soln. of pure **I** in acetone dilut with the buffer soln. to give a soln. containi 20 μg of **I** per ml. A. O. Jones

1761. **Determination of 2,6-dichloro-4-nitr aniline in ditranil and some fungicidal formulatio by infra-red spectroscopy.** P. G. Marshall (Standar Dept., Boots Pure Drug Co. Ltd., Beeston, Nottin ham, England). *Analyst*, 1960, **85**, 681–683.- Mixed standards in $CHCl_3$ containing 0·356 0·396% of 2,6-dichloro-4-nitroaniline (the acti principle of ditranil), 0 to 0·04% of 2-chloro-nitroaniline and 0·008% of chloranil (the tw impurities present in ditranil) are prepared. Th sample (40 mg) is dissolved in 10 ml of CHC The i.r. spectra are recorded over the range 11 to 1095 cm^{-1} and the base-line absorptions at 114 1128 and 1113 cm^{-1} are measured. From th measurements of the standard soln. a calibratio graph is plotted from which the amount of eac component of the sample is found. With the dus Allisan, the sample (1 g) is shaken with eth (60 ml) for 30 min., the extract is evaporated, th residue is dissolved in 20 ml of $CHCl_3$ and the i. spectrum is recorded at 1145 cm^{-1} as described f ditranil. The spectrum of a standard soln. co taining 40 mg of the active ingredients in 20 ml $CHCl_3$ is recorded similarly. The percentage active ingredient present is obtained by mult plying the ratio of the base-line absorption of th sample to that of the standard by 4. For a 50% dispersible powder, 0·4 g is shaken with 100 ml $CHCl_3$ for 30 min. The clear supernatant soln. used for the i.r. measurement as described f Allisan, with the same standard soln.
A. O. Jones

See also Abstracts—**1750**, Analysis of chlor methylphenoxypropionic acids. **1660**, K in plant **1662**, Mg in plants. **1667**, V and Co in plant **1676**, Carotenoids in plants. **1693**, Cystine i feeding-stuffs. **1720**, Methoxychlor in milk. **1774** F in plants.

5.—GENERAL TECHNIQUE AND APPARATUS

General

1762. **Interchangeable conical ground glass joint** British Standards Institution (2 Park Stree London, W.1). B.S. 572:1960. 11 pp.—Thi edition takes into account recommendations unde discussion in the International Organisation fo Standardisation and aims to ensure that standar joints made in Britain shall be interchangeable wit those produced abroad.

1763. **Small rotary sampler and preliminary stud of its use.** R. W. M. Hawes and L. D. Mulle (Chemical Engng Div., A.E.R.E., Harwell, England A.E.R.E. Report AERE–R3051, 1960. 22 pp.- The sampler (illustrated) was designed for use wit dry ore products. It was intended that the devic which continuously divides the ore into 20 parts should have a performance superior to that of th microriffle with which it was critically compare and a statistical analysis of the results suggeste

this was so. The difficulties of designing quate comparison experiments are considered.
G. J. Hunter

764. **New hydrogen sulphide apparatus.** C. J. Nieuwenburg and J. W. L. van Ligten (Techn. v., Delft, Netherlands). *Chem. Weekbl.*, 1960, (40), 550.—A pressure-regulating vessel con- ing machine oil is described, which is mounted H_2S cylinders in order to avoid losses of H_2S ing the opening and closing of the cylinder.
M. J. Maurice

765. **Notes on the preparation and construction silver reductor columns.** J. I. Dinnin (U.S. l. Survey, Washington, D.C.). *Anal. Chim. a*, 1960, **23** (3), 295–296 (in English).—It is gested that silver used in reductor columns uld be prepared from mixtures of the copper- uced and electro-deposited types. This allows rates to be chosen at will. Pulverised metallic er and metal platelets may be incorporated with antage. A suitable column design is described illustrated that has a siphon type of outflow e with the outlet above the level of the metal . Drying out of the column can thus be avoided.
H. M.

766. **Density hydrometers for use in milk. t 1. Apparatus.** British Standards Institution Park Street, London, W.1). B.S. 734: Part 1: 0. 18 pp.—Specifications are given for three es of hydrometer.

767. **Improvements in or relating to burettes.** torius-Werke A.-G. Brit. Pat. 854,998; date l. 31.3.59. Germany, date appl. 24.4.58.— a burette having a motor-actuated piston, the ult of the displacement of the piston in the nder is transmitted to an indicating device, , a counting device and/or a digit printing ice, the gearing for actuating the piston and gearing for the indicating device being separated from the other.
J. M. Jacobs

768. **An automatic recording titrimeter and its sible uses.** S. Wolf. *Öst. ChemZtg*, 1960, **61**), 292–298.—The various electrical methods of ficating the end-point of a titration are discussed, l the use of the Metrohm Potentiograph E–336 a recorder of titration curves when such tech- ues are employed is described. With this trument both normal titration curves and erential curves can be recorded and it may be d with aq. and non-aq. soln. When the instru- nt is used alone, acid - base and $AgNO_3$ titra- ns can be followed, and amperometric and stant-current potentiometric titrations are sible. The Potentiograph can be coupled with Coulometer E–211 for coulometric titrations and h a Konduktoskop E–165 for conductimetric rk. Typical titration curves are presented.
H. M.

769. **Apparatus for use in chemical analysis.** ternational Analyser Co. Brit. Pat. 848,125; te appl. 31.1.58. Belgium, date appl. 1.2.57.— e apparatus, which enables periodic chemical alyses of an aq. soln. to be made in a completely tomatic way, comprises a vessel for containing aq. soln. to be analysed, a Mariotte vessel to ntain the titrating soln. and a collecting vessel contain a mixture of the soln. and connected to aq. soln. vessel by a discharge pipe from the ter into which opens an outlet pipe from the rating soln. vessel. The aq. soln. vessel communicates with the air and is connected to the collecting vessel by a siphon which holds back the soln. in the aq. soln. vessel as long as the level of the aq. soln. is below the level of the top of the siphon. The siphon automatically empties the aq. soln. vessel each time this level is reached, and this constant vol. is delivered at such a rate of flow that a vacuum is created in the discharge pipe, so that in operation a constant vol. of titrating soln. is added, at every discharge, to the aq. soln. flowing through the siphon, the mixture being then collected, for analysing purposes, in the collecting vessel.
J. M. Jacobs

1770. **The 'magnification procedure' in colorimetric ultra-micro titrations with photo-electric determination of the end-point.** J. Mika and B. Szopory (Tech. Univ. f. Heavy Ind., Miskolc, Hungary). *Mikrochim. Acta*, 1960, (5–6), 729–748 (in German).—The method involves the comparison of the colour of a test soln. treated with reagent in a capillary cell with that of a soln. of relatively large vol., obtained by titrating a known vol. of reagent with a standard soln. of the test material. The method is applied to the determination of Ti with H_2O_2, the error on 2 to 30 μg of TiO_2 being $< \pm 1\%$. Sources of error are discussed in detail.
J. P. Stern

1771. **Methods of liquid - liquid partition.** G. J. Henning (Inst. Verw. Geneeskr. Arom. Gewassen, Groningen, Netherlands). *Pharm. Weekbl.*, 1960, **95** (20), 645–658.—Multiplicative partition methods (Craig distribution) are discussed and the extraction apparatus of von Metzsch (*Chem.-Ing.-Tech.*, 1950, **22**, 209) is described. Two examples of the application of this technique are given, *viz*, the separation of rutin, hyperin and quercitrin and the analysis of the compounds from hops that are pptd. by lead salts. Discontinuous partition, with repeated addition of the compounds to be analysed, is also discussed and the purification of streptomycin is given as an example. Finally, columns for continuous extraction are described.
M. J. Maurice

1772. **Liquid nitrogen level filler control.** V. E. Schupbach (Goodyear Atomic Corp., Portsmouth, Ohio, U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1536.—The device, the main component of which is an inverted Hoke HGP valve, is simple and efficient. The operation depends on the temp., and hence the pressure, of a 2-g charge of propane in the sensing element.
C. B. Baines

1773. **The influence of viscometer design on non-Newtonian measurements.** R. McKennell (Ferranti Ltd., Manchester, England). *Anal. Chem.*, 1960, **32** (11), 1458–1463.—The design described offers uniform shearing conditions and a flexible automatic flow-curve recorder. The effect of shear-rate variation for Newtonian fluids in conventional viscometers is discussed, and various methods for eliminating variations in shear rate, and hence for eliminating the necessity for applying corrections to flow curves for different types of non-Newtonian behaviour, are outlined. The newly designed viscometer is compared with certain industrial viscometers.
C. B. Baines

1774. **Improved multiple all-glass distillation apparatus for determination of fluorine in plant samples.** R. F. Brewer and G. F. Liebig, jun. (Univ. of California Citrus Exp. Station, Riverside, U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1373.—With

the apparatus described, and under routine conditions, the quant. transfer of 5 to 2000 μg of F in 350 ml of distillate is achieved in ≃ 50 min. The reproducibility of results is excellent, and the results of duplicate determinations usually agree to within ± 5%. K. A. PROCTOR

1775. **Improvements in and relating to apparatus for the measurement of the hydrazine content in a solution.** Cambridge Instrument Co., Ltd. [Inventor: H. W. Holy]. Brit. Pat. 848,525; date appl. 20.3.58.—The apparatus comprises a gold (or other noble metal) electrode and a silver - silver chloride electrode immersed in a chloride electrolyte (10% aq. KCl). The arrangement is such that when a soln. under test, *e.g.*, a sample of boiler-feed water, is introduced into the cell, the hydrazine is oxidised on the surface of the gold electrode, thereby causing a current proportional to the hydrazine concn. of the soln. to flow between the gold and the silver - silver chloride electrodes. J. M. JACOBS

1776. **New rapid vacuum fusion apparatus for determining oxygen in titanium and steels.** L. C. Covington and S. J. Bennett (Process Res. Div., Titanium Metals Corp. of America, Henderson, Nev.). *Anal. Chem.*, 1960, **32** (10), 1334–1337.—The apparatus described consists of a resistance-heated furnace, a power supply, a duplicate analytical system and an evacuation system. One operator can complete 75 oxygen determinations in 8 hr. with good precision and accuracy.
K. A. PROCTOR

1777. **Simple apparatus for differential thermal analysis.** M. Vaniš and O. Koráb (Inst. Chem. Technol., Prague). *Silikáty*, 1960, **4** (3), 266–270.—Thermo-elements carrying samples are placed in a vertical four-channel tube. Two methods can be used—(*i*) the samples are placed in a furnace which is then heated; (*ii*) the samples are moved into a heated furnace by means of a transferring device. J. ZÝKA

1778. **Simple construction of apparatus for automatic differential thermal analysis.** V. Šatava and Z. Trousil (Inst. Chem. Technol., Prague). *Silikáty*, 1960, **4** (3), 272–277.—A description of an automatic programmed-temp. controller and an apparatus for the direct recording of DTA curves, in which semi-conducting elements are used, is given.
J. ZÝKA

1779. **Differential thermal analysis and simultaneous gas analysis.** C. B. Murphy, J. A. Hill and G. P. Schacher (Gen. Engng Lab., General Electric Co., Schenectady, N.Y., U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1374–1375.—The equipment described permits the simultaneous collection of gas generated during thermal decomposition and its registration as a peak on the thermogram obtained on differential thermal analysis. K. A. PROCTOR

See also Abstract—**1340**, Progress in equipment and tools for micro-analysis.

Chromatography, ion exchange, electrophoresis

1780. **Relationship between R_F and the composition of the eluent isopropyl alcohol - hydrochloric acid (1 : 1).** T. Oncescu and S. Zwiebel (C. I. Parhon Univ., Bucharest). *Zhur. Anal. Khim.*, 1960, **15** (4), 409–412.—R_F values were determined for a large number of ions with the eluent isopropyl alcohol - HCl (1 : 1), with various normalities HCl, and these were generally in good agreeme with those obtained by Carvalho (*Anal. Abst* 1958, **5**, 351), with n-butanol - HCl (1 : 1) as eluen Sodium can be satisfactorily separated from K b descending chromatography with 4N-HCl in th eluent mixture ($\Delta R_F = 0{\cdot}09$); Rb and Cs are we separated with 12N-HCl, group-II elements wit 10N-HCl, Zn and Cd with 9N-HCl, Hg from Z and Cd with low HCl concn., Tl from Al or In wit 5·5N-HCl, Be and Al with 10N-HCl, Ca and with 12N-HCl, Zn and Th with 6N-HCl (ΔR_F = 0·11); As^{3+} and As^{5+} with 10N-HCl; U, Th an Zr with 5N-HCl; and Ni and Co with 10N-HC There is a parallel relationship between the sol bility of the metal chlorides or chloro-complex and the R_F values. K. R. COOK

1781. **Stable diazo-salts for chromatograph spray reagents.** I. A. Pearl and P. F. McCoy (Ins Paper Chem., Appleton, Wis., U.S.A.). *Ana Chem.*, 1960, **32** (11), 1407–1410.—The colou developed when 20 phenolic compounds were ea sprayed on paper with 30 commercially availab stabilised diazo-salts of aromatic amines a reported. The salts used are stable for years.
G. BURGER

1782. **New electromechanical integrator, especial for gas-chromatographic analysis.** M. Mohnke an K. Renker (Dtsch. Akad. Wissenschaft., Berlin *Chem. Tech., Berlin*, 1960, **12** (8), 493–494.—A fu description is given of the construction and use an automatic germanium-diode-actuated phot electric device for measuring and recording th band areas of the chromatograms, and hence givi automatically the amounts of the components the gas or vapour. Results are accurate to with 4%. H. L. WHITEHEAD

1783. **Transfer device for gas chromatograph** A. Kreuchunas (DWG Cigar Corp., Detroit, Mich U.S.A.). *Chemist Analyst*, 1960, **49** (3), 82–83. The apparatus illustrated allows the transfer very small fractions to glass capillary tubes f storage, etc. Full constructional details are give P. D. PARR-RICHARD

1784. **An injection system for gas chromat graphy.** W. W. Nawar, F. M. Sawyer, E. Beltran and I. S. Fagerson (Dept. of Food Techno Univ. of Mass., Amherts, U.S.A.). *Anal. Chem* 1960, **32** (11), 1534.—The system described is easi constructed in the laboratory at small cost fro readily available materials. A fairly high vacuu can be applied to allow rapid transfer of hig boiling volatiles, with no appreciable sample lo to almost all types of gas-chromatographic equi ment. It is efficient, particularly in application food volatiles. Recoveries of 90% have be attained with a test mixture containing ni components boiling from 27° to 173·5°.
C. B. BAINES

1785. **Apparatus for sampling a fluid and injecti it into a gas-chromatography column.** Distille Co., Ltd. [Inventor: C. W. Munday]. Brit. Pa 850,132; date appl. 24.6.58.—The apparatus co prises first and second (perpendicular) lines throu which the fluid to be sampled, and the eluting g passing into the column, respectively, can flo and a linear tube laterally interrupting these lin A slidable and non-rotatory plunger, tigh mounted within the tube, is pierced by a passa the inlet and outlet of which are adapted to conn and be aligned with the interrupted first or seco

, and which is shaped to promote stream-lined
of fluid through it. Both the plunger and tube
constructed of quartz and their surfaces in
tact are ground. J. M. Jacobs

786. **Gas - liquid chromatography. Pro-
mmed-temperature control of the capillary
umn.** R. Teranishi, C. C. Nimmo and J. Corse
ric. Res. Service, U.S. Dept. of Agric., Albany,
if., U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1384–
6.—The method described has been developed
the examination of complex natural products
ling over a wide range. Thin-walled stainless-
el capillary tubing is used, giving the advantage
low heat capacity and high heat conductance.
amples described are a complex hydrocarbon
xture and a strawberry flavouring. An argon
isation detector is used. C. B. Baines

787. **Improvements relating to vapour fracto-
ters.** Perkin-Elmer Corp. Brit. Pat. 849,408;
e appl. 30.4.59; U.S.A., date appl. 2.6.58.—
preparatory column for a vapour fractometer
nprises a number of hollow tubes of equal length
cked with an inert material, *e.g.*, diatomaceous
th, supporting a film of liquid substrate developer,
., dodecyl phthalate. The tubes are provided
their opposite ends with common end-caps
ming a sealing fit with the open ends of the
bes and embodying inlet and outlet connections
the assembly of tubes and connecting conduits
tween the ends of adjacent tubes. One of the
d caps is displaceable in relation to the other to
able the number of tubes connected in series
tween the inlet and the outlet to be adjusted so
to vary the effective column length, and give
e most expeditious and desirable separation of
mple mixtures in accordance with their particular
ysical characteristics as related to vapour
ctometry techniques. J. M. Jacobs

1788. **Factors affecting the use of gas - liquid
romatography for the separation of large samples.
e column dimensions.** W. J. de Wet and V.
etorius (Central Tobacco Res. Sta., Kroondal,
ansvaal, S. Africa). *Anal. Chem.*, 1960, **32** (11),
96–1399.—The efficiency of a chromatographic
lumn has been investigated and theoretical and
perimental evidence shows that, for a given
lumn efficiency, the max. sample vol. increases
either the column diameter or the column length
increased. C. B. Baines

1789. **Applications of Carbowax 400 in gas
romatography for extreme aromatic selectivity.**
R. Durrett (Shell Oil Co., Refinery Lab., Houston,
ex., U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1393–
96.—This stationary phase is very useful for the
lective retention of aromatic hydrocarbons relative
paraffinic hydrocarbons. Two gas-chromato-
aphic methods for the analysis of petroleum
mples are modified to use Carbowax 400 as the
ationary phase. The determination of aromatic
drocarbons in aviation petrol is described, and
sults from 10 aviation blends containing known
olumes of benzene and toluene show standard
eviations of 0·02% of benzene and 0·05% of
luene, indicating the method to be more accurate
an the F.I.A. method for total aromatics. The
etermination of hydrocarbon impurities in petro-
um benzene and toluene is also described, and
sults show the method to be superior to the ASTM
attwinkel reagent test. C. B. Baines

1790. **Molten salt mixtures as liquid phases in
gas chromatography.** W. W. Hanneman, C. F.
Spencer and J. F. Johnson (California Research
Corp., Richmond, U.S.A.). *Anal. Chem.*, 1960, **32**
(11), 1386–1388.—This new column permits opera-
tion in the temp. range 150° to 400°. Mixtures
of the nitrates of Na, K and Li are used as the
stationary phase supported on firebrick. Tables
are presented showing the resolution of various
types of organic compounds. Optimum resolution
is attained with samples of 1 μl; this sample size is
handled easily with thermal conductivity detectors
and has the advantage of linear response.
C. B. Baines

1791. **Use of a carbon monoxide flame detector
in gas chromatography.** C. B. Baddiel and C. F.
Cullis (Dept. of Chem., Imperial College, London,
England). *Chem. & Ind.*, 1960, (37), 1154–1155.—
Halogenated methanes were separated on a Johns
Manville C22 firebrick column (360 cm × 4 mm)
impregnated with 20% of dinonyl phthalate, and
measured with a flame detector. The use of H as
the carrier gas led to corrosion by hydrogen halides
and to quenching of the flame. These difficulties,
together with the variable flow rate due to the
high diffusivity of H, were avoided by the use of
CO burnt at a wide jet 2 mm in diam. The flow
rate of CO was 35 ml per min. and that of air
400 ml per min. In general, the peak area of each
compound was linearly related to the sample
pressure, but the peak due to chlorotrifluoro-
methane consisted of two positive parts separated
by a negative trough; this is ascribed to combustion
variation when different parts of the molecule were
burnt. R. M. S. Hall

1792. **A highly sensitive katharometer and its
application to the measurement of ethylene and
other gases of biological importance.** S. P. Burg
and J. A. A. Stolwijk (Harvard Univ., Cambridge,
Mass., U.S.A.). *J. Biochem. Microbiol. Technol.
Engng*, 1959, **1**, 245–259.—The design and con-
struction of the katharometer are described. It
has been found to possess more than 100 times
the sensitivity of conventional thermal-conductivity
instruments. The equipment is adaptable to the
precise determination of ethylene and other gases
by gas chromatography. The construction of the
instrument and the gas-flow circuit are illustrated.
Routine analyses of biologically produced ethylene
were conducted and for this purpose the instrument
has proved to be about 10^4 times as sensitive as
other methods (Young *et al.*, *Amer. J. Botany*,
1959, **35**, 814). Factors influencing the sensitivity
of katharometers are considered. Chem. Abstr.

1793. **A new type of selective ion exchanger.**
J. Seidl and J. Štamberg (Res. Inst. Synth. Resins
and Lacquers, Pardubice, Czechoslovakia). *Chem.
& Ind.*, 1960, (38), 1190–1191.—A new type of
chelate resin has been prepared from fluorone
derivatives and formaldehyde. It is selective for
Ge even in conc. HCl and is not affected by the
presence of As and Fe. N. E.

1794. **Micro-electrophoresis on cellulose acetate
membranes.** B. W. Grunbaum, P. L. Kirk and
W. A. Atchley (Cancer Res. Inst., Univ. of Cali-
fornia, Berkeley, U.S.A.). *Anal. Chem.*, 1960, **32**
(10), 1361–1362.—The use of cellulose acetate
supporting medium (Kohn, *Clin. Chim. Acta*, 1958,
3, 450) in conjunction with an apparatus previously
described (Grunbaum and Kirk, *Anal. Chem.*, 1960,
32, 564) is recommended as giving results greatly

superior to those obtained with filter-paper. Staining with Ponceau S (C.I. Acid Red 9) (0·2% in 3% trichloroacetic acid) is preferred as being rapid (10 min.), and easily washed out with 5% acetic acid. H. F. W. KIRKPATRICK

1795. **Ion separation on a thread.** J. Mahon and A. A. Benedetti-Pichler (Brooklyn College, New York, U.S.A.). *Mikrochim. Acta*, 1960, (5–6), 831–835 (in English).—A more sensitive alternative to the ring-oven method for separating metal cations is described. After extraction of the thiocyanates of Fe^{3+}, Co and Zn with ethyl ether, those of Cu and Ni are complexed with pyridine and extracted with $CHCl_3$. Each extract is chromatographed by dipping a piece of acetone-extracted sewing thread into the soln. and the coloured zones are examined by white or u.v. light under the microscope. The colourless $Zn(SCN)_4^{2-}$ is identified by its fluorescence reaction with morin. Distinct zones are given by 3 μg of each cation. P. D. PARR-RICHARD

Optical

1796. **Oscillating flat-specimen holder for an X-ray powder camera.** J. D. Wilkinson and L. D. Calvert (Div. of Appl. Chem., N.R.C., Ottawa, Canada). *J. Sci. Instrum.*, 1960, **37** (10), 399–400.—A simple and readily demountable holder for flat specimens, designed to facilitate the direct comparison of films, is described. Details of construction are given. G. SKIRROW

1797. **Causes and control of matrix effects in spectrographic discharges.** A. J. Frisque [Res. and Develop. Dept., Standard Oil Co. (Indiana), Whiting, Ind., U.S.A.]. *Anal. Chem.*, 1960, **32** (11), 1484–1488.—Changes in the discharge temperature resulting from matrix changes have been measured and found to explain the observed intensity-ratio changes. A correction factor based on the discharge temperature provides a significant improvement in analytical accuracy. New requirements for matching line pairs are defined, and the choice of suitable internal standards is discussed. C. B. BAINES

1798. **Elimination of anion interferences in flame spectroscopy. Use of ethylenedinitrilotetra-acetic acid.** A. C. West and W. D. Cooke (Baker Lab., Cornell Univ., Ithaca, N.Y., U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1471–1474.—In the method described, EDTA is used to minimise or eliminate interference caused by a variety of anions, including phosphate and sulphate, in the determination of Ca, Mg, Co, Cu, Cr and Mn at low concn. The addition of EDTA also enhances the flame emission and greatly stabilises the intensity. The mechanism of this is discussed. Some of the experimental variables in flame spectroscopy are examined, and an instrument is described that has an absolute stability of $\pm$ 2% over a period of 2 months. C. B. BAINES

1799. **Connection between the sensitivity of the determination and the parameters of the spectrographic apparatus.** Z. G. Fratkin. *Zavod. Lab.*, 1960, **26** (8), 971–973.—The effects of various factors, plate contrast, resolution, dispersion, etc., on the sensitivity of spectrographic analysis are discussed. G. S. SMITH

1800. **Modification of Beckman DK-2 spectrophotometer circuitry and operation.** E. Cogan, H. Freund and P. Romas (Albany Metallurgy Res. Center, Bur. of Mines, U.S. Dept. of the Interio Albany, Oreg.). *Anal. Chim. Acta*, 1960, **23** (3 294–295 (in English).—By modifying the workin procedure the pen travel may be increased so a to give enhanced peak heights. Modification the circuit, when combined with the above pr cedure, will give even greater pen travel. Th new procedures are described and are stated to giv little increase in noise level. H. M.

1801. **Correction of fluorescence spectra an measurement of fluorescence quantum efficiency** C. A. Parker and W. T. Rees (Admiralty Materia Lab., Holton Heath, Poole, Dorset, England *Analyst*, 1960, **85**, 587–600.—Commercial spectro fluorimeters record "apparent fluorescence emissio and excitation spectra," both of which in som regions of the spectrum are distorted versions of th true spectra, and may be reproducible for the sam instrument but differ considerably from result obtained with other instruments of different type The origin of fluorescence spectra is discusse theoretically. The measurement of true excitatio and emission spectra is described. The advantage of using true fluorescence spectra are discussed, an the use of true emission spectra to determin fluorescence quantum efficiencies is described Standardisation of published spectrofluorimetri data should be achieved when possible. Result for six compounds that fluoresce in the visible regio of the spectrum are presented. A. O. JONES

1802. **Effect of organic nitrogen solvents upo specific rotation.** J. Rosin and C. J. William (Merck and Co. Inc., Rahway, N.J.). *J. Amer Pharm. Ass., Sci. Ed.*, 1960, **49** (9), 622–624.—Dat are presented to show that aniline, butylamine 2-aminoethanol, isoquinoline, pyridine and espec ially quinoline may be used as solvents to enhanc the optical rotation of 32 alkaloids, steroids an other drugs, thus increasing the accuracy of identifi cation and assay. The soln. should be protecte from strong light during preparation and handlin and should be kept for not more than 24 hr. befor measurement. A. R. ROGERS

See also Abstract—**1353**, Computer for comple mentary tristimulus colorimetry. **1451**, Apparatu for determination of Pb.

Electrical

1803. **Alternating-current polarography an tensammetry.** H. H. Bauer (Fac. of Agric., Univ. Sydney, Australia). *J. Electroanal. Chem.*, 1960 **1** (5), 363–378 (in English).—The use of a.c. polaro graphy and tensammetry in chemical analysis an in interpreting reaction mechanisms is discussed with many examples taken from the literature Surface-active agents, WO_4^{2-} and other non reducible compounds can be determined by measur ing the height of either of the tensammetric wave produced by periodic adsorption/desorption of th species at the electrode/soln. interface. A calibra tion curve is necessary because peak current *vs* concn. curves are non-linear. (104 references.) W. J. BAKER

1804. **Rapid polarography.** S. Wolf (Metrohm A.-G., Herisau, Switzerland). *Angew. Chem.* 1960, **72** (13), 449–454.—By use of a mercury elec trode of controlled dropping time of <1 sec., th complete recording time of a polarogram may b reduced by a factor of five. The limiting current i almost completely independent of the potential

oscillations are greatly reduced in amplitude. ima of the second type are not observed.
A. R. Rogers

05. **Vector polarograph.** S. B. Tsfasman. *d. Lab.*, 1960, **26** (7), 888–889.—A sinusoidal age of fixed amplitude between 4 and 40 mV applied simultaneously with the polarising age. Since the capacity current is in quadrature the active component of the electrolytic ent a vector-polarogram showing the relation veen current and polarising voltage, in which effect of the capacity current does not appear, be obtained. Apparatus for this purpose is ribed. It has a sensitivity of $\simeq 10^{-7}$M and is h simpler in construction than the Barker re-wave polarograph. G. S. Smith

806. **New electrode for cathode-ray polarography.** C. Penketh (I.C.I. Ltd., Heavy Org. Chem. Div., ngham, England). *J. Appl. Chem.*, 1960, **10** 324–328.—The polarographic cell consists of concentric mercury-pool electrodes. The r electrode forms a rising cathode, in which cury under a constant head of pressure is wed to rise and overflow for a fixed time after potential sweep in order to renew the surface. sensitivity of the electrode is proportional to ace area, and, with a diameter of 12 mm, $\simeq 0{\cdot}01$ m. of In^{3+} can be detected and $\simeq 0{\cdot}05$ p.p.m. be determined. The peak heights are repro- ible and are linearly proportional to the concn. he ion. J. H. Waton

807. **Simple micro-coulometric arrangement for rography using the three-electrode system.** J. ek (Polarogr. Inst., Czech. Acad. Sci., Prague). *Electroanal. Chem.*, 1960, **1** (5), 416–421 (in lish).—In the cell described and illustrated, a hite "lead" from a "Koh-i-Noor" HB pencil ns the working anode, the lower mercury rvoir acts as reference electrode (no current, st. potential), whilst the third electrode is the pping-mercury type. The vol. of sample soln. 0·2 ml and the voltage between the dropping trode and the mercury reservoir is measured a volt-ohm meter having an input impedance 0 to 100 MΩ. The cell gave satisfactory results completely irreversible reactions, *viz*, reduction H^+ in 0·1M-KCl, of $[Co(NH_3)_6]^{3+}$ in 0·1M-HCl, of HN_3 in 14M-H_2SO_4. Sources of error are cussed. W. J. Baker

808. **Purification of neutral salt solutions for arographic basal electrolytes from ultra-small ounts of heavy-metal impurities.** E. N. Vinodova and K. Iobst (M. V. Lomonosov Moscow te Univ.). *Zavod. Lab.*, 1960, **26** (7), 796–797.—n. kept in containers of amber glass tend to lose vy-metal impurities. Colloidal sulphide of iron the glass gradually hydrolyses, and the S^{2-} eract with Zn, Pb, Cd and Cu present in the soln. en a soln. of KCl (0·1N) containing Pb (10^{-5}M), ($3{\cdot}5 \times 10^{-5}$M), Cd ($2{\cdot}5 \times 10^{-5}$M) and Cu ($2 \times$ $^{-5}$M), was passed at a rate of 5 ml per min. through olumn (25 cm × 5 cm) of powdered amber glass trace of the heavy metals could be detected arographically. G. S. Smith

809. **A potentiograph, and automatic registering ation apparatus.** K. Hädicke (Dtsch. Metrohm isting & Co. KG., Essen, Germany). *Fette, f., Anstrichmitt.*, 1960, **62** (7), 584–586.—An instrument is described which registers changes in pH, mV or current against the volume of non-aqueous soln. added. It is suitable for titrations involving small potential changes at the end-point in dark media or emulsions. Titration curves are given. The error for pure fatty acids is ±1%, that of a mixture of fatty acids with up to 7 components is ±3%. I. Dickinson

1810. **Hydrogen electrodes for pH determination and potentiometric titration.** L. N. Davÿdov. *Lab. Delo*, 1960, **6** (4), 54–57.—The micro-electrode described consists of two parts: a pump and a special electrode consisting of two non-communicating chambers (*a* and *b*). Chamber *a* has an internal diameter of 0·4 cm, height 1 cm and a capacity of 0·1256 ml, and is suitable for holding $\simeq 0{\cdot}06$ ml of test liquid. Chamber *b* serves for the saturation by H of the main platinum-black surface. The passage of H into the chamber is controlled by rotating the piston; thus a rotation of 180° cuts off the H supply. The electrode is filled with test soln. by raising the piston. The test soln. is saturated with H by shaking the electrode for 3 to 5 min. Comparative data are given for the pH determination of whole blood, and of phosphate buffer soln. with and without quinhydrone, obtained with different types of hydrogen electrode. A diagram is given of a hydrogen electrode for the determination of pH in potentiometric macro-titration. K. R. Cook

1811. **The relation of catalyst acidity to the electrode potential recorded in an automatic titration.** R. O. Clark, E. V. Ballou and R. T. Barth (Gulf Res. and Dev. Co., Pittsburgh, Pa., U.S.A.). *Anal. Chim. Acta*, 1960, **23** (2), 189–195 (in English).—The surface acidity of catalysts is determined by potentiometric titration of catalyst slurries in methyl cyanide with a standard n-butylamine soln. The method has been successfully adapted for use with an automatic titrimeter and the titration curves produced are in agreement with data obtained from titrations with indicators.
G. S. Roberts

1812. **Electrolytic re-oxidation after current reversal in chronopotentiometry.** C. Furlani and G. Morpurgo (Chem. Inst., Univ., Trieste, Italy). *J. Electroanal. Chem.*, 1960, **1** (5), 351–362 (in English).—A mathematical procedure is given for determining the transition time (t') of the re-oxidation that occurs after current reversal at the end of an electrolytic reduction in chronopotentiometry, when the reduced species is unstable and is decomposing according to a first-order reaction. Assuming semi-infinite linear diffusion and equal diffusion coeff. for the reduced and oxidised species, a simple equation relates the ratio of the transition times (t, t') of reduction and re-oxidation with the rate const. (K) of the reaction, independently of all other parameters. The value of K can be determined directly by measurement of $t':t$ experimentally. The validity of the equation has been confirmed for the chronopotentiometric reduction and subsequent re-oxidation of 2×10^{-3}M-Fe^{3+} in $\simeq$3M-KOH and 0·1M-triethanolamine, with 0·04 to 0·07M-hydroxyammonium chloride as regenerant for the oxidised species. The method should be useful for the study of electrode processes accompanied by rapid reactions, or for determining labile intermediates during, for example, reduction of metal complexes that undergo previous dissociation.
W. J. Baker

1813. **Simple device for accurate time-measurement in coulometric analysis.** F. Vorstenburg and A. W. Löffler (Techn. Univ., Delft, Holland). *J. Electroanal. Chem.*, 1960, **1** (5), 422–423 (in English).—The error inherent in the use of a stop-clock during a short electrolysis is minimised by substituting an electronic counter which counts the pulses of a transistorised crystal oscillator having const. frequency f. When a diode is included there is no inertia during switch-on and -off, whilst the max. counting error is $1/f$ sec. per measuring period. The circuit is shown. W. J. BAKER

1814. **Automatic measurements and computations for radiochemical analyses.** J. N. Rosholt, jun., and J. R. Dooley, jun. (U.S. Geological Survey, Denver, Colo.). *Anal. Chem.*, 1960, **32** (9), 1093–1098.—Methods of measurement and computation have been developed for the analysis of natural radioactivity which facilitate the large-scale determination of radioactive sources for geochemical studies. K. A. PROCTOR

1815. **Electron affinity spectroscopy—a new method for the identification of functional groups in chemical compounds separated by gas chromatography.** J. E. Lovelock and S. R. Lipsky (Dept. of Internal Med., Yale Univ. Sch. of Med., New Haven, Conn., U.S.A.). *J. Amer. Chem. Soc.* 1960, **82** (2), 431–433.—A simple ionisation chamber is described, containing a small radium source of α-particles. The voltage applied to the chamber may be varied between 0 and 100. The presence of organic vapours in a He gas stream causes an increase or decrease in the intensity of the current flowing through the chamber. It has been found that, for most organic compounds, the electron affinity, which governs the effect in the ionisation chamber, is related to the major functional group. By altering the applied voltage it has been shown that the effects of various types of compound change from positive to negative at particular voltages. By choosing a definite voltage and placing the ionisation chamber in the outlet stream of a gas chromatograph it is possible to distinguish between compounds of different basic structure (*e.g.*, ketones in the presence of hydrocarbons). It is suggested that this device would be of value in qualitative gas chromatography. At present Ar cannot be used as carrier gas, but suggestions are made for modifications which might permit its use. It should then be possible to carry out quantitative measurements with a conventional Ar detector, and to classify the compounds by subsequent passage of the gas stream through the detector described. In its present form the apparatus can be used with N, H or He. T. R. ANDREW

1816. **Interpretation of γ-ray scintillation spec from fission-product mixtures.** D. H. Peirs (A.E.R.E., Harwell, England). *Brit. J. Ap Phys.*, 1960, **11** (8), 346–353.—The γ-ray scintillati spectra of fission-product mixtures can be int preted quant. by reference to standard spect calculated for the 22 products of the simultaneo slow-neutron fission of ^{235}U. In this way, differenc from the standard series can be readily related differences in fission-product yields or disintegrati rates. The decay-scheme and crystal-efficien data for the standard spectra are tabulated, a the 36 activities (arranged in order of γ-ray energy together with their corresponding variations peak-height during 1 to 28 days, are listed. A rap method of half-life analysis, based on the composi peak-height decay curve, for use when peaks a inadequately resolved by energy is describe Because of an anomaly in spectrometer resolutio the calculated standard peak-height efficiency mu be corrected by an empirical relationship dete mined for the scintillation crystal used in t spectrometer. W. J. BAKER

1817. **Automatic compensation of dead time pulse-analysis equipment.** D. F. Covell, M. Sandomire and M. S. Eichen (U.S. Naval Radi logical Defense Lab., San Francisco, Calif.). *An Chem.*, 1960, **32** (9), 1086–1090.—A timing devi for the compensation of dead time in an Argonn type 256-channel analyser has been evaluated a compensation was found to be satisfactory over range of 500 to 24,000 counts per sec. Becau of its simplicity the method is more satisfacto than ultra-fast detectors and circuits. The tec nique is applicable to problems of dead time equipment generally. K. A. PROCTOR

1818. **Improved sample-loading technique f solid-source mass spectrometry.** R. E. Powe (Atomic Weapons Res. Estab., Aldermasto Berks., England). *J. Sci. Instrum.*, 1960, **37** (7 252–253.—The sample is loaded by evaporation an aq. soln., dropwise, on a tungsten filamen Heat is provided by holding a soldering iron contact with the filament supports. By this mea the sample is confined to the centre of the filamen G. SKIRROW

See also Abstracts—**1342**, Review of use of nucle magnetic resonance. **1355**, Chronopotentiometi with current reversal. **1356**, **1357**, Chronopotenti metric potential - time curves. **1370**, Extracti radiometric titration. **1451**, Apparatus for dete mination of Pb. **1732**, Apparatus for detectir air contamination.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	A
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
Colour Index	C.I.
concentrated	conc.
concentration	concn.
constant	const.
crystalline	cryst.
crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
electromotive force	e.m.f.
equivalent	equiv.
ethylenediaminetetra-acetic acid	EDTA
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecule-e, -ar	mol.
normal (concentration)	N
optical rotation	α_l^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

The chemical nomenclature used in the abstracts is that recommended by The Chemical Society ("Handbook for Chemical Society Authors", The Chemical Society, London, 1960), and may differ from that used in the original articles.

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